LIBBY ASBESTOS SUPERFUND SITE, OPERABLE UNIT 3

DATA SUMMARY REPORT: 2007 TO 2013

Revision 2 – January 2015

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LIBBY ASBESTOS SUPERFUND SITE
OPERABLE UNIT 3

DATA SUMMARY REPORT: 2007 TO 2013

REVISION LOG:

Revision No.	Date	Description
0	11/4/13	--- *[Included data summaries for investigations from 2007 to 2011]*
1	7/21/14	• Revised to include 2012 data summaries: o Section 2.5 & Section 3.4, Phase V Part A surface water & sediment o Section 2.6 & Section 3.5, Phase V Part B surface water & sediment o Section 6.3 & Section 8.4, "near mine" commercial logging bark/duff & ABS o Section 8.3, Kootenai River recreational ABS (Phase V Part A) o Section 9.3, amphibian sediment toxicity test o Section 9.4, in-stream fish toxicity test o Section 10.5, resident trout population study o Section 10.6, amphibian field population study • Updated Section 12 to include laboratory audits, QC reviews, validation efforts conducted in 2012 o Added Section 12.6 to document water analysis issues in 2012 • Minor editorial changes incorporated
2	1/20/15	• Revised to include 2013 data summaries: o Section 5.2, Amphitheater removal o Section 6.2, Phase I bark/duff replicate TEM analyses o Section 8.5, supplemental TEM analysis of ABS air samples o Section 8.6, Souse Gulch wildfire ABS o Section 9.4.2, repeat eyed egg toxicity test • Added missing duff transect elevation figures (transect 135° and 255°) • Added Phase IV-B "free fiber" surface water results (Section 2.4) • Updated Section 12 to include laboratory audits, QC reviews, validation efforts conducted in 2013 • Minor editorial changes incorporated

Approved by: _____ Date: 2/2/15

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Acronyms and Abbreviations

%	percent
<	less than
≤	less than or equal
>	greater than
ABS	activity-based sampling
AOC	Administrative Order on Consent
ATSDR	Agency for Toxic Substances and Disease Registry
ATV	all-terrain vehicle
BTC	Bobtail Creek
BTT	tributary to Bobtail Creek
BCS	biological condition score
BERA	baseline ecological risk assessment
BMI	benthic macroinvertebrate
BTAG	Biological Technical Assistance Group
CB&I	CB&I Federal Services
cc	cubic centimeters
cc^{-1}	per cubic centimeter
CDM Smith	CDM Federal Programs
COC	chain-of-custody
CSF	Close Support Facility
DO	dissolved oxygen
DQO	data quality objective
DSR	data summary report
EDD	electronic data deliverable
ELI	Energy Laboratories, Inc.
EMSL	EMSL Analytical, Inc.
EPA	U.S. Environmental Protection Agency
EPH	extractable petroleum hydrocarbon
ESAT	Environmental Services Assistance Team
FEL	Fort Environmental Laboratories
FSDS	field summary data sheets
ft^3/sec	cubic feet per second
GO	grid opening
Golder	Golder Associates, Inc.
gpm	gallons per minute
Grace	W.R. Grace Company
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDR	HDR Engineering, Inc.
HEI-AR	Health Effects Institute-Asbestos Research
HQS	habitat quality score
HSI	habitat suitability index
H&S	health and safety
in.	inches
ISO	International Organization for Standardization

KDC	Kootenai Development Corporation
KR	Kootenai River
LA	Libby amphibole asbestos
LRC	lower Rainy Creek
MDEQ	Montana Department of Environmental Quality
MFL	million fibers per liter
MFWP	Montana Fish Wildlife and Parks
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliters
mm	millimeter
mm^2	square millimeters
Ms/cm^2	million structures per square centimeter
Ms/g	million structures per gram
MWH	MWH Americas, Inc.
NFG	National Functional Guidelines
NIST	National Institute of Standards and Technology
NSY	Noisy Creek
NVLAP	National Voluntary Laboratory Accreditation Program
ORP	oxidation reduction potential
OSHA	Occupational Safety and Health Administration
OSU	Oregon State University
OU	operable unit
OU3	Operable Unit 3
PAH	polycyclic aromatic hydrocarbon
PCB	polychlorinated biphenyl
PCM	phase contrast microscopy
PCME	phase contrast microscopy-equivalent
PE	performance evaluation
PERL	Parametrix Environmental Research Laboratory
PLM	polarized light microscopy
PLM-VE	polarized light microscopy, visual area estimation
PLM-Grav	polarized light microscopy, gravimetric
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
QATS	Quality Assurance Technical Support
RBP	rapid bioassessment protocol
Remedium	Remedium Group, Inc.
RI	remedial investigation
ROM	Record of Modification
RPM	regional program manager
SAP	sampling and analysis plan
Shaw	Shaw Environmental, Inc.
Site	Libby Asbestos Superfund Site
SOP	standard operating procedure
SPF	Sample Preparation Facility

SPP	soil preparation plan
SRC	SRC, Inc.
SVL	snout-vent length
SVOC	semi-volatile organic compound
TAT	turn-around analysis
TEM	transmission electron microscopy
TLI	TLI Solutions, Inc.
TOC	total organic carbon
µg/L	micrograms per liter
µm	micrometer
URC	lower Rainy Creek
USFS	U.S. Forest Service
UV	ultraviolet
VOC	volatile organic compound
WVB	Whitlock-Vibert box
YOY	young of the year

1 Introduction

1.1 Project Background

Libby is a community in northwestern Montana that is located 7 miles southwest of a vermiculite mine that operated from the 1920s until 1990. The mine began limited operations in the 1920s and was operated on a larger scale by the W.R. Grace Company (Grace) from approximately 1963 to 1990. Vermiculite from the mine contains a form of asbestos referred to as Libby amphibole (LA). This site is of potential concern to the U.S. Environmental Protection Agency (EPA) primarily because historic mining, milling, and processing of vermiculite at the site are known to have caused releases of LA to the environment, and inhalation exposure to asbestos is known to increase the risk of cancer and non-cancer effects in humans (Agency for Toxic Substances and Disease Registry [ATSDR] 2001).

Epidemiological studies revealed that workers at the mine had an increased risk of developing asbestos-related lung disease (McDonald *et al.* 1986, 2004; Amandus and Wheeler 1987; Amandus *et al.* 1987; Whitehouse 2004; Sullivan 2007). Additionally, radiographic abnormalities were observed in 17.8 percent (%) of the general population of Libby including former workers, family members of workers, and individuals with no specific pathway of exposure (Peipins *et al.* 2003; Whitehouse *et al.* 2008; Antao *et al.* 2012; Larson *et al.* 2010, 2012a, 2012b). Although the mine has ceased operations, historic or continuing releases of LA from mine-related materials could be serving as a source of ongoing exposure and risk to current and future residents and workers in the area. Based primarily on these concerns, EPA listed the Libby Asbestos Superfund Site (Site) on the National Priorities List in October 2002.

Given the size and complexity of the Site, EPA divided the site into eight operable units (OUs). Operable Unit 3 (OU3) is defined as the property in and around the Zonolite Mine owned by Grace or Grace-owned subsidiaries (excluding the former Screening Plant) and any area (including any structure, soil, air, water, sediment, or receptor) impacted by the release and subsequent migration of hazardous substances and/or pollutants or contaminants from such property, including, but not limited to, the mine property, the Kootenai River and sediments therein, Rainy Creek, Rainy Creek Road and areas in which tree bark is contaminated with such hazardous substances and/or pollutants and contaminants. Because the final boundary of OU3 cannot be determined without data on the extent of mine-related contamination, for the purposes of conducting investigations, EPA established an initial study area for OU3, as shown in **Figure 1-1**.

Kootenai Development Corporation (KDC), a subsidiary of Grace, owns the mine and land surrounding the mine. EPA has entered into an Administrative Order on Consent (AOC) with Respondents Grace and KDC. The designated Project Coordinator for Grace and KDC is Remedium Group, Inc. (Remedium). Under the terms of the AOC, the Respondents are performing a remedial investigation (RI) in OU3, under EPA oversight, in order to characterize the nature and extent of environmental contamination and to collect data to allow EPA to

evaluate risks to humans and ecological receptors from mining-related contaminants in the environment.

1.2 Overview of OU3 Sampling Activities

Sampling in support of the OU3 RI is being performed in several phases. Sampling and analysis activities performed as part of each phase are conducted in accordance with EPA-developed program-specific sampling and analysis plans (SAPs) and/or quality assurance project plans (QAPPs). An overview of the various sampling programs are discussed briefly below.

Phase I of the RI was performed in the fall of 2007 in accordance with the *Phase I Sampling and Analysis Plan for Operable Unit 3* (EPA 2007). The primary goal of the Phase I investigation was to obtain preliminary data on the levels and spatial distribution of LA and non-asbestos chemicals that might have been released to the environment in the past as a consequence of the mining and milling activities at the Site.

Phase II of the RI was performed in the spring, summer, and fall of 2008. Phase II was composed of three parts, as follows:

- Part A (EPA 2008a) focused on the collection of data on the levels of LA and non-asbestos chemicals in surface water and sediment, as well as site-specific toxicity testing of surface water using rainbow trout.

- Part B (EPA 2008b) focused on the collection of data on LA levels in ambient air samples collected near the mined area, and on the collection of data on LA and non-asbestos chemicals in groundwater.

- Part C (EPA 2008c) primarily focused on the collection of aquatic habitat and community data and site-specific toxicity tests to support the ecological risk assessment at the Site. This SAP also included the collection of data on the levels of LA and non-asbestos chemicals in surface water and sediment at selected reference stations.

Phase III of the RI was performed in the spring, summer, and fall of 2009 in accordance with the *Phase III Sampling and Analysis Plan for Operable Unit 3* (EPA 2009a). Phase III included the collection of activity-based air samples during simulated recreational visitor activities in the forested area, as well as the collection of a variety of ecological community and habitat metrics in support of the ecological risk assessment.

Phase IV of the RI was performed in 2010 and 2011. Phase IV was composed of two parts, as follows:

- Part A of the Phase IV SAP (EPA 2010a) was performed in the summer and fall of 2010. Part A focused on the collection of additional activity-based air samples during simulated recreational visitor, wood harvesting, forest management, and firefighting activities to support the human health risk assessment.

- Part B of the Phase IV SAP (EPA 2011a) was performed in the spring, summer, and early fall of 2011. Part B focused on the collection of additional data on LA levels in surface water to support the ecological risk assessment. Data collection efforts also included sampling to better characterize the habitat suitability of site streams for fish.

Phase V of the RI was performed in 2012. Part A of this sampling program (EPA 2012a) included the collection of LA levels in surface water, sediment, and activity-based air samples during simulated recreational visitor activities on the Kootenai River. Part B of this sampling program (EPA 2012b) included a series of ecological studies to support the ecological risk assessment, including an amphibian toxicity test, an amphibian field assessment, in-stream caged fish studies, a resident fish lesion study, and a fish tissue burden assessment. Due to issues with one of the caged fish studies (i.e., the eyed egg study), it was repeated in 2013 (EPA 2013a).

In September 2012, activity-based air samples were collected during authentic commercial logging activities (EPA 2012c) to support the human health risk assessment.

In 2013, two different sampling efforts were conducted. The first sampling effort was to collect soil samples following removal activities at the Amphitheater due to the observation of asbestos-containing vermiculite waste (MWH 2012). The second sampling effort was an opportunistic air sampling study that was conducted during a small wildfire that occurred near the Souse Gulch recreation area.

1.3 Document Purpose and Organization

As noted above, this document is a data summary report (DSR) for OU3 that presents results from sampling efforts conducted between 2007 and 2013; an overview of these sampling activities is provided in **Table 1-1**. **Table 1-2** summarizes the SAPs/QAPPs, along with any associated document modifications, for each sampling investigation. Although portions of these results have been presented previously (as part of the Phase II, III, and IV SAPs), this document provides a summary of all results in a single comprehensive report. This DSR is amended annually to summarize results collected in the preceding field season. Data collected in 2014 will be summarized in the next annual update. This document is only intended to summarize the results of each sampling program; the interpretation of these results or an evaluation of data adequacy to support risk management decision-making is beyond the scope of this report.

In addition to this introduction, this report is organized into the following sections:

Section 2 – Surface Water

Section 3 – Sediment

Section 4 – Groundwater

Section 5 – Soil and Mine Waste from the Mined Area

Section 6 – Soil, Duff Material, and Tree Bark from the Forested Area

Section 8 – Ambient Air

Section 9 – Activity-Based Sampling (ABS) Air

Section 10 – Aquatic Toxicity Tests

Section 11 – Aquatic Habitat and Community Surveys

Section 12 – Small Mammal Surveys

Section 13 – Quality Assurance/Quality Control

Section 14 – References

All tables and figures cited in the text are provided at the end of the report. Appendices are provided electronically.

2 Surface Water

Surface water samples were collected at OU3 as part of the Phase I, Phase II (Part A), Phase IV (Part B), and Phase V sampling programs. Surface water samples were analyzed for a broad suite of analytes, including LA and non-asbestos chemicals. The following sections summarize the surface water field data for each sampling program conducted from 2007 to 2013. Detailed summaries of results for asbestos and for non-asbestos chemicals in surface water samples are provided in **Appendix B** and **Appendix C**, respectively.

2.1 Phase I (2007)

2.1.1 Sampling Design

The objective of the Phase I sampling program was to collect surface water data to obtain a preliminary characterization of the nature and extent of potential surface water contamination related to historical mining, milling/processing, and mine-waste disposal operations. In addition, the Phase I sampling program also conducted a visual survey to identify and sample any springs where groundwater discharge was present and any seeps emanating from mine waste disposal areas.

Figure 2-1 identifies the locations where surface water samples were collected in Phase I. Station identifiers are summarized in **Table 2-1**. All surface water samples were collected in October 2007. All surface water samples were analyzed for LA, metals/metalloids, petroleum hydrocarbons, anions, and other water quality parameters. In addition, a broad suite of analyses were performed for samples collected at two locations: the tailings impoundment toe drain (TP-TOE1) and lower Rainy Creek downstream of the confluence with Carney Creek (LRC-2). These locations were selected because they appeared to have the best potential of characterizing releases from the mine. The additional analyses for surface water included polychlorinated biphenyls (PCBs), pesticides, herbicides, gross alpha/gross beta, volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), polycyclic aromatic hydrocarbons (PAHs), and cyanide. At the time of sample collection, field measurements of several water quality metrics, including temperature, pH, specific conductance, dissolved oxygen (DO), oxidation/reduction potential (ORP), turbidity, and stream discharge, were measured using portable field meters.

All surface water sampling was conducted by MWH Americas, Inc. (MWH) (a contractor to Remedium). Detailed information on the Phase I field sampling effort, including all associated field documentation, is provided in the *Phase I Field Sampling Summary Report* (MWH 2007).

After water samples were collected in the field, the samples for asbestos analysis were hand-delivered to the EMSL Analytical, Inc. laboratory in Libby, Montana (EMSL-Libby) for filtration. (Note: No treatment of the water was performed prior to the filtration.) The resulting filters were analyzed by EMSL-Libby for total LA by transmission electron microscopy (TEM). Filters

were prepared and analyzed using EPA Method 100.2 (EPA 1994), with modified counting procedures as described in Libby Laboratory Modification #LB-000020.

Analyses of non-asbestos chemicals in surface water were performed by Energy Laboratories, Inc. (ELI) in Billings, Montana (a contractor to Remedium).

Detailed analytical results for all Phase I surface water samples (asbestos and non-asbestos) and field-collected water quality metrics are provided in the OU3 master project database (see **Appendix A**). The following sections summarize these results.

2.1.2 Non-Asbestos Results

Table 2-2 presents summary statistics on the detection frequency and concentration of non-asbestos analytes detected in water samples analyzed as part of the Phase I sampling program. As seen, a number of inorganic constituents (metals, anions, and nitrogen compounds) were detected in water, as were several indicators of petroleum hydrocarbons; but no VOCs, SVOCs, PCBs, or PAHs were detected. Metals were detected more frequently and at higher concentrations in seeps than in other surface water reaches. Additionally, several metals, including chromium, lead, nickel, vanadium, and zinc were reported as detected only in seep samples. Petroleum hydrocarbons were detected in locations within the Fleetwood Creek reach and in two seeps (CCS-1 and CCS-14). Benzene was detected in only one seep (CSS-14).

2.1.3 Asbestos Results

Table 2-3 summarizes the results of the analysis of surface water (and seeps) for LA (based on total[1] structures and structures longer than 10 micrometers [µm]). Water concentrations are expressed in terms of million fibers per liter (MFL). As seen, detected concentrations of total LA ranged widely (more than three orders of magnitude), from less than 0.1 to 125 MFL.

Figure 2-2 is a map that displays the spatial pattern of total LA results in surface water. The highest levels were observed in samples located in ponds or impoundments, including the tailings impoundment, the Mill Pond, and the pond on Fleetwood Creek, as well as from several seeps along the south side of the mined area. Levels in lower Rainy Creek (below the Mill Pond) tended to be relatively low. A sample collected just upstream of the confluence of Rainy Creek and the Kootenai River (LRC-6) was non-detect.

2.1.4 Field Measurement Results

Field data measurements collected at surface water locations sampled during the Phase I study included temperature, pH, specific conductance, DO, ORP, and turbidity. **Table 2-4** summarizes field data measurements for surface water. Temperature varied by only a few degrees at stations within stream reaches. Additionally, pH did not vary significantly within each stream reach; the lowest pH was measured in Carney Creek and the highest pH was observed in lower Rainy Creek. At most locations, DO concentrations were below 14 milligrams per liter (mg/L);

[1] This includes LA structures 0.5 µm and longer with an aspect ratio (length:width) of 3:1 or greater.

surprisingly, DO was above 20 mg/L at three of the seep locations. Turbidity was generally higher in the pond and seep samples than in stream samples.

Table 2-5 presents stream discharge measurements collected at a number of stations in the Rainy Creek watershed. As seen, flows were generally low (usually less than about 0.2 cubic feet per second [ft^3/sec]), especially in Fleetwood Creek, Carney Creek, and the upper reaches of Rainy Creek. Flows in the lower reach of Rainy Creek were slightly higher, with an average flow rate of 0.5 ft^3/sec.

2.2 Phase II, Part A (Spring-Fall 2008)

2.2.1 Sampling Design

Data from Phase I sampling program provided information on the concentrations of LA and other non-asbestos chemicals in surface water for a single sampling event (in October 2007). Because concentrations of chemicals in surface water may vary over time, especially in cases where there are large fluctuations in flow (e.g., during spring runoff), the objective of the Phase II Part A sampling program was to collect additional data to characterize the temporal and spatial patterns of LA and non-asbestos chemicals in surface water at OU3.

The Phase II Part A sampling program consisted of two monitoring efforts – one for the Rainy Creek watershed and one for the Kootenai River. Stations included in the Phase II Part A sampling program are identified in **Table 2-6**. The Rainy Creek watershed monitoring effort was split into several "elements" as follows:

> *Element 1: Seasonal Monitoring* – The purpose of this element was to measure stream flow and contaminant concentrations of LA and non-asbestos chemicals in surface water at the stations sampled in Phase I to characterize levels during spring and summer flow conditions. Four additional sampling locations – UTP, TP-Overflow, URC-1A, and CC-Pond – were also sampled (see **Figure 2-3**). Two rounds of sampling were completed – one in June 2008 and one in September 2008. All surface water samples were analyzed for LA, metals/metalloids, petroleum hydrocarbons, anions, and other water quality parameters. In addition, a broad suite of analyses were performed for samples collected at the tailings impoundment toe drain (TP-TOE1) and lower Rainy Creek downstream of the confluence with Carney Creek (LRC-2). As noted previously, these locations were selected because they appeared to have the best potential of characterizing releases from the mine. The additional analyses for surface water include PCBs, pesticides, herbicides, gross alpha/gross beta, VOCs, SVOCs, and cyanide. At the time of sample collection, field measurements of several water quality metrics, including temperature, pH, specific conductance, DO, ORP, turbidity, and stream discharge, were measured using portable field meters.
>
> *Element 2: Spring Runoff Monitoring* – The purpose of this element was to monitor stream flow and surface water LA concentrations at selected stations within the Rainy Creek watershed during the rising and falling limbs of the spring snowmelt-runoff

hydrograph. **Figure 2-4** identifies the stations that were sampled as part of Element 2. Surface water samples were collected weekly at each station beginning at the onset of rising stream flows in response to snowmelt, continuing through the spring high-flow season, and ending after the seasonal peak in flow is observed on Rainy Creek (from early April through mid-June 2008). All surface water samples were analyzed for LA. At the time of sample collection, stream flow was measured.

Element 3: Summer and Fall Monitoring – The purpose of this element was to provide on-going information on LA concentrations and stream flow rates downstream of asbestos sources within the Rainy Creek watershed. Two lower Rainy Creek stations were sampled as part of Element 3 - the station below Carney Creek (LRC-2) and the station near its discharge to the Kootenai River (LRC-6). Surface water samples were collected every two weeks at each station, beginning in mid-June and ending in mid-August 2008. All surface water samples were analyzed for LA. At the time of sample collection, stream flow was measured.

Element 4: Continuous Precipitation and Flow Monitoring – The purpose of this element was to collect continuous data on precipitation and stream flow. To accomplish this, a rain gauge was placed at the meteorological station on the mine site and permanent flumes were installed at LRC-2, LRC-6, and CC-2.

Element 5: Collection of Surface Water for Toxicity Testing – The purpose of this element was to collect site surface water for use in site-specific toxicity tests. This element is discussed further in Section 9.1.

The Phase II Part A sampling program also collected surface water samples in the Kootenai River. **Figure 2-5** provides a map of the surface water sampling locations in the Kootenai River. These locations were selected to provide surface water LA concentrations upstream and downstream of Rainy Creek and to include river locations with the greatest potential for elevated LA concentrations due to transport via Rainy Creek. Although the planned study included sampling during both high flow and low flow conditions, due to safety concerns for sampling personnel during high flow, samples were only collected under low flow conditions. All surface water samples were analyzed for LA.

All surface water sampling was conducted by MWH. Detailed information on the Phase II Part A field sampling effort, including all associated field documentation, is provided in the *Phase II Field Sampling Summary Report* (MWH 2009).

After water samples were collected in the field, the samples for asbestos analysis were hand-delivered to EMSL-Libby for filtration (Note: No treatment of the water was performed prior to the filtration). The resulting filters were then analyzed at EMSL-Libby or sent to the EMSL laboratory in Beltsville, Maryland (EMSL-Beltsville) for analysis of total LA by TEM. Filters were prepared and analyzed in basic accordance with the International Organization for Standardization (ISO) method 10312:1995(E) (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications.

Analyses of non-asbestos chemicals in surface water were performed by ELI.

Detailed analytical results for all Phase II Part A surface water samples (asbestos and non-asbestos) and field-collected water quality metrics are provided in the OU3 master project database (see **Appendix A**). The following sections summarize these results.

2.2.2 Non-Asbestos Results

Table 2-7 presents summary statistics on the detection frequency and concentration of non-asbestos analytes detected in water samples analyzed as part of the Phase II Part A sampling program. As seen, a number of inorganic constituents (metals, anions, and nitrogen compounds) were detected in water, but VOCs were not detected. The only hydrocarbon detected was total extractable hydrocarbon at seep CCS-8 in the June 2008 sampling event. At the two locations (TP-TOE1 and LRC-2) analyzed for the broader suite of analytes, only gross alpha and gross beta were detected.

2.2.3 Asbestos Results

Tables 2-8 through **2-11** summarize LA surface water concentrations (based on both total structures and structures longer than 10 μm) for the Phase II Part A sampling program for each element, respectively.

Element 1. **Table 2-8** summarizes the LA results for surface water (and seeps) sampled in June and September as part of Element 1. As seen, detected concentrations of total LA ranged widely (more than four orders of magnitude), from 0.1 to over 1,000 MFL. The highest levels were observed in samples located in ponds or impoundments, including the pond on Fleetwood Creek and the tailings impoundment, as well as from several seeps along the south side of the mined area. However, it is possible that the higher levels noted in these samples could have been attributable to higher amounts of sediment in these samples as a consequence of sample collection methods. Total LA levels in upper Rainy Creek were usually non-detect. Total LA levels in lower Rainy Creek (below the Mill Pond) tended to be less than 9 MFL, with higher concentrations generally reported during the June sampling event.

Element 2. **Table 2-9** summarizes LA results for the 11-week surface water sampling effort conducted as part of Element 2. The greatest fluctuation in total LA concentration was observed at the tailings impoundment, with total LA ranging from over 1,000 MFL in week 2 to about 3 MFL in week 11. In lower Rainy Creek, total LA concentrations fluctuated from one to two orders of magnitude over the 11-week period, with highest concentration and flows observed during week 7 (measured on May 19, 2008). **Figure 2-6** presents surface water flow and total LA concentration graphically for stations LRC-1, LRC-2, and LRC-6. As shown in this figure, there is a clear correlation between flow and concentration in lower Rainy Creek, when flow is high, concentration is high. **Figure 2-7** illustrates surface water flow and total LA concentrations graphically for stations (URC-1A, URC-2) in upper Rainy Creek and station FC-2 in Fleetwood Creek. As seen, flow and LA concentrations seem to correlate at FC-2, but not at upper Rainy Creek Stations. **Figure 2-8** shows surface water flow and total LA concentrations for tailings

impoundment stations, TP-TOE1 and TP-Overflow. This figure indicates that LA concentrations tend to be higher when flow rates are higher at these locations. Graphs of all stations are presented in **Appendix B**.

Element 3. **Table 2-10** summarizes LA results for the eight surface water samples collected from mid-June to mid-August at LRC-2 and LRC-6 as part of Element 3. The average total LA concentration for both locations tended to be similar (about 3 MFL). **Figure 2-6** shows surface water flow and total LA concentration graphically for stations LRC-2 and LRC-6 for Element 3 and includes LRC-1 from Element 2. As seen, for LRC-1 and LRC-6 total LA concentrations appear to be higher when flow rates are higher; however, flow rates were not available for all samples making it impossible to establish an empirical relationship. Whereas total LA concentrations at LRC-2 do not correlate with flow rates and appear to level out over mid-June to mid-August.

Kootenai River. **Table 2-11** summarizes LA results for surface water collected from the Kootenai River under low flow conditions (August). Total LA structures were observed in surface water collected from two of the stations located downstream of Rainy Creek, but concentrations tended to be low (≤ 0.1 MFL). The surface water sample collected upstream of Rainy Creek was non-detect.

2.2.4 Field Measurement Results

Tables 2-12 through **2-14** summarize field data measurements collected at surface water locations during each element of the Phase II Part A sampling events in 2008. Measurements were collected to evaluate spring and summer flow conditions during Element 1, spring runoff conditions during Element 2, summer and fall conditions in Element 3, and low flow (fall) conditions in the Kootenai River. Field measurements included: temperature, pH, specific conductance, DO, ORP, and turbidity. As would be expected, temperatures were lower and turbidity was higher in surface water samples collected in the spring than later in the year. Normally temperature varied by only a few degrees at stations within stream reaches during each sampling period. Generally higher temperatures were measured in the tailings impoundment and ponds. Additionally, pH did not vary significantly within each stream reach or sampling event. At most locations, DO concentrations ranged between 6 mg/L and 14 mg/L.

2.3 Phase II, Part C (Fall 2008)

2.3.1 Sampling Design

The Phase II Part C sampling program primarily focused on the collection of aquatic habitat and community data and site-specific toxicity tests needed to support the ecological risk assessment at the site. In addition, this sampling program also included the collection of surface water samples at two selected aquatic reference stations. Two of three candidate aquatic reference stations were sampled (see **Figure 2-9**) – Noisy Creek (NSY-R1) and a tributary to Bobtail Creek (BTT-R1). BTT-R1 was sampled in preference to the other candidate aquatic reference station on Bobtail Creek (BTC-R1).

Surface water samples were collected from each aquatic reference station in October 2008. All surface water samples were analyzed for LA, metals/metalloids, water quality parameters, pesticides, herbicides, and SVOCs. At the time of sample collection, field measurements of several water quality metrics, including temperature, pH, specific conductance, DO, ORP, turbidity, and stream discharge, were measured using portable field meters.

All surface water sampling was conducted by Parametrix (a contractor to Remedium). Detailed information on the Phase II Part C field sampling effort, including all associated field documentation, is provided in the *Final Data Report for the Autumn 2008 Aquatic Data Collection Program* (Parametrix 2009a).

After water samples were collected in the field, the samples for asbestos analysis were hand-delivered to EMSL-Libby for filtration. (Note: No treatment of the water was performed prior to the filtration.) The resulting filters were then analyzed by EMSL-Libby for total LA by TEM. Filters were prepared and analyzed in basic accordance with ISO 10312:1995(E) (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications.

Analyses of non-asbestos chemicals in surface water were performed by ELI.

Detailed analytical results for all Phase II Part C surface water samples (asbestos and non-asbestos) and field-collected water quality metrics are provided in the OU3 project database (see **Appendix A**). The following sections summarize these results.

2.3.2 Non-Asbestos Results

Table 2-15 presents a summary of non-asbestos analytes detected in water samples analyzed as part of the Phase II Part C sampling program. As seen, a number of metals were detected in water and slight differences between the two reference stations were observed. Dissolved cadmium was detected at NSY-R1 but not at BTT-R1. Pesticides, herbicides, and SVOCs were not detected above reporting limits at either location.

2.3.3 Asbestos Results

Table 2-16 summarizes the total LA surface water results for the aquatic reference stations. As seen, total LA was not detected in surface water samples from reference areas.

2.3.4 Field Measurement Results

Table 2-17 presents field data measurements for aquatic reference stations collected during the Phase II Part C sampling event. Field measurements included: temperature, pH, specific conductance, DO, and ORP. There appear to be some differences in reference station water characteristics based on the limited set of field measurements. Temperature was higher at BBT-R1 and conductivity was about 25 percent lower than the value measured at NSY-R1. However, DO was similar at both stations and was about 11 mg/L. pH was not measured at NSY-R1 due to an instrument malfunction, pH at BTT-R1 was about 8.

2.4 Phase IV, Part B (2011)

Part B of Phase IV focused on the collection of additional site surface water data needed to support the ecological risk assessment. Data collection efforts included sampling and analysis of site surface waters to characterize temporal LA concentrations, as well as efforts to better characterize the habitat suitability of site streams for fish. The sampling design and results of the habitat assessment are discussed in Section 10.4. The surface water sampling design and results are presented below.

2.4.1 Sampling Design

Because surface water samples collected as part of the Phase I and Phase II sampling investigations may have been influenced by fibers clumping and adhering to sampling container walls, asbestos concentration values observed in these samples are uncertain. The objective of the Phase IV Part B sampling program was to collect additional surface water data to better characterize temporal LA concentrations in surface water at OU3, as well as provide more reliable data on LA water concentrations for evaluating exposures to aquatic receptors.

The Phase IV Part B sampling program consisted of regular monitoring of LA concentrations in surface water at a subset of sampling locations. This included locations where permanent flumes had been placed, including two stations in Rainy Creek (LRC-2 and LRC-6) and one station in Carney Creek (CC-2), and at the tailings impoundment (TP) (see **Figure 2-3**). These stations were selected because Lower Rainy Creek is the chief reach of concern for fish, and these stations are downstream of potential primary sources of asbestos, including the tailings disposal area (LRC-2), sediments deposited along lower Rainy Creek (LRC-6), and site seeps and ponds (CC-2). Station TP (in the tailings impoundment) was selected because it is representative of waters to which amphibians may be exposed. In order to characterize the levels of LA in surface water as a function of flow, time (season), and location, weekly sampling was conducted from mid-April (prior to the onset of rising stream flows in response to snowmelt) to July 2011 at each station, followed by bi-weekly sampling after spring flows decreased through the end of September 2011.

Whenever surface water samples were collected, the in-stream temperature, pH, specific conductance, DO, ORP, and turbidity were also measured using portable field meters. In addition, continuous flow monitoring was performed at LRC-2, LRC-6, and CC-2.

Previous studies indicate that LA fibers in natural waters (i.e., rivers, streams) may be present as "free fibers" (i.e., not associated with organic matter) or as "fiber clumps" (in the water or adhered to the container walls). Because it is not known what fiber form may be important for evaluating exposures to aquatic receptors, concentrations of LA in surface water were measured based on both free fibers and total fibers (i.e., free fibers + fiber clumps).

Total fiber samples. The collection of water samples for total fiber analysis used standard sampling methods (i.e., a grab sample was collected using a peristaltic pump from the flowing water). After water samples were collected in the field, the samples for asbestos analysis were

hand-delivered to EMSL-Libby for treatment (ozonation/ ultraviolet [UV] light) and sonication in accordance with the procedures in EPA Method 100.1 prior to filtration. Use of ozonation/ UV treatment prior to filtration addresses potential fiber clumping/wall adherence issues (EPA 2011a). The resulting filters were then analyzed at EMSL-Libby for total LA by TEM. Filters were analyzed in basic accordance with the ISO 10312:1995(E) (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications.

Free fiber samples. Water samples for the analysis of free fibers were collected using an OU3-specific sampling technique (SOP No. 3A), which involved collecting a sample of water in a syringe and then immediately filtering the sample through a syringe filter disk in the field. The filter cassettes were then transported to Hygeia Laboratories, Inc. (Hygeia) in Sierra Madre, California for preparation and analysis. Filters for the measurement of free LA fibers were analyzed using the same counting protocols as for total fibers, except that only free fibers (i.e., fibers not associated with organic material clumps) were counted. The analyst also noted the occurrence of large organic LA-containing clumps and reported this on the EDD.

All surface water sampling was conducted by MWH. A detailed report, providing all the field information and documentation for the Phase IV Part B field sampling effort has not been prepared.

2.4.2 Asbestos Results

Table 2-18 summarizes the surface water results of the total fiber samples collected during the Phase IV Part B investigation; concentrations are shown based on both total LA and LA longer than 10 μm in length. As seen, LA was detected in all but one sample and values ranged widely, up to 276 MFL. The highest concentrations of LA in surface water were in samples from lower Rainy Creek and were observed in the May sampling events when flows were highest. Concentrations of LA greater than 10 μm in length were detected in all but three samples and values ranged up to 55 MFL.

Table 2-19 summarizes the results of the free fiber samples; the corresponding total fiber sample results are also presented for comparison. As shown, only the filters collected as part of the first three rounds of sampling were analyzed. With two exceptions, free fiber concentrations tended to be lower than the total fiber concentrations, but higher than concentrations based on LA longer than 10 μm in length. Clumps of fibers were observed in nearly all water samples.

Figure 2-10 displays the temporal distribution of total LA results along with flow measurements (for LRC-2, LRC-6, and CC-2). As seen, LA concentrations are higher when flow rates are higher and the highest flow rates were measured in May. However, some exceptions are noted, for example at LRC-2, LA concentrations increased in late July and August, but no corresponding increase in flow was noted. Additionally the elevated LA concentration observed at CC-2 on September 20, 2011 prompted the collection of an opportunistic sample on November 9, 2011. As seen on **Figure 2-10** and in **Table 2-18** LA concentrations in surface water at CC-2 in November were significantly lower than in September.

2.4.3 Field Measurement Results

Table 2-20 summarizes field data measurements collected at surface water locations sampled during the Phase IV Part B which include: temperature, pH, specific conductance, DO, ORP, and turbidity. Temperature data shows a clear temporal trend for all stations, with cooler temperatures in the spring and peaks during the hottest summer months; pH did not vary significantly within each stream reach or sampling event. At most locations, DO concentrations were below 6 mg/L. For CC-2 and LRC-6, turbidity was generally highest in the spring.

2.5 Phase V, Part A (2012)

Part A of the Phase V investigation focused on characterizing the potential nature and extent of asbestos in surface water in the Kootenai River downstream of the confluence with Rainy Creek. This investigation was designed to collect surface water data for the Kootenai River to address limitations identified with the existing data. As specified in the SAP/QAPP (EPA 2012d), previous surface water data were collected during low flow conditions (no data were available under high flow conditions) and the collected water samples were not treated with ozone or ultraviolet light prior to filtration, which potentially biased the reported concentrations low due to asbestos fibers clumping and adhering to the walls of the sample containers. In addition, previous Kootenai River water collection efforts only included a limited number of sampling locations.

The overall objective of this investigation was to provide information to better characterize levels of LA in the Kootenai River under both high flow and low flow conditions and to provide data that can be used to determine if concentrations of LA in surface water in the Kootenai River downstream of the confluence with Rainy Creek are above a level of human health concern.

2.5.1 Sampling Design

Relative loading from Rainy Creek to the Kootenai River is dependent upon numerous factors. As shown in previous studies (see **Figures 2-6** and **2-7**), LA concentrations in Rainy Creek tend to be higher in the spring when flow is highest. The timeframe for the Phase V Part A sampling events spanned seasonal high flow conditions (i.e., spring run-off) from Rainy Creek to the Kootenai River so as to capture peak loading to the Kootenai River. Because Rainy Creek is expected to be the principal source of LA to the Kootenai River, samples were collected both upstream and downstream of the confluence with Rainy Creek to characterize the potential effect of Rainy Creek on the river. Surface water grab samples were collected from stations UKR-0, KR-1, KR-4, KR-5, and LRC-6 (see **Figure 2-11**) over an eight week period from late April 2012 through mid-June 2012, at a frequency of one sample per week. In order to evaluate low flow conditions, one surface water grab sample was also collected from each of these locations in mid-September 2012**.**

In addition to the samples listed above, five locations (KR-14, KR-15, KR-16, UKR-1, and UKR-3[2]) (see **Figure 2-11**) were sampled once in late May 2012 and once in September 2012 during high and low flow periods, respectively. Surface water grab samples were collected from the bank at all locations. Additionally, at locations KR-14 and KR-16, river transect surface water samples (four samples at equally-spaced intervals across the river) were collected using a flat-bottom powerboat on the same day that the grab sample was collected from the bank.

All surface water sampling was conducted by MWH (a contractor to Remedium). Detailed information on the Phase V, Part A field sampling effort, including all associated field documentation, is provided in the *Phase V Remedial Investigation Field Sampling Summary Report* (MWH 2013).

After the water samples were collected in the field, the samples were hand-delivered to EMSL-Libby for filtration and analysis. Prior to filtration, all samples underwent ozonation/ultraviolet treatment and sonication in basic accordance with the techniques in EPA Method 100.2 (EPA 1994), as modified by Libby Laboratory Modification LB-000020A.

Resulting filters from high-flow samples were used to prepare grids using the grid preparation techniques described in Section 9.3 of ISO 10312:1995(E). These grids were initially examined by rapid turn-around analysis (TAT) by EMSL-Libby using TEM in basic accordance with the procedures described in an OU3-specific method modification (TEM_WATER_Mod1)[3]. In brief, grids were examined by TEM in basic accordance with the counting rules specified in ISO 10312:1995(E), but the analyst was not required to report structure-specific attributes for each countable structure observed. Rather, the analyst was only required to report the total number of countable LA structures for each grid opening examined, which was then used to estimate the total LA water concentration.

Originally, the intent was to utilize the results of the rapid TAT TEM analyses to identify the three-week time period when concentrations of LA in the Kootenai River were the highest. Then, all samples collected within this three-week time period were to be analyzed by the "standard" TEM method and other samples outside of this three-week time period would be archived for possible future analysis. However, based on a preliminary evaluation of the rapid TAT results, EPA determined that, with the exception of a subset of samples from locations LRC-6 and UKR-0, all samples would undergo standard TEM analysis. For locations LRC-6 and UKR-0, samples from the last three rounds of sampling during high flow conditions were archived.

During the "standard" TEM analyses, the prepared grids were examined by EMSL-Libby, EMSL-Cinnaminson, or EMSL-Denver using TEM in basic accordance with the procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory

[2] This station was identified in as UKR-2 in the Phase II, Part A investigation.
[3] A copy of this method modification is available in the Libby OU3 eRoom.

Modifications LB-000016, LB-000020, LB-000029, LB-000066, LB-000067, and LB-000085. Surface water samples were not analyzed for non-asbestos chemicals.

Beginning on April 25, 2012, continuous flow measurements were collected using ISCO® automated flow recorders installed at LRC-6, LRC-2 and CC-2. The data collected at LRC-6 was monitored to ensure that the surface water samples were representative of high flow conditions within Rainy Creek.

As noted in the *Phase V Remedial Investigation Field Sampling Summary Report* (MWH 2013) measurement of water quality parameters (e.g., pH, specific conductivity, etc.) and stream discharge were not performed at surface water sample locations in accordance with the SAP/QAPP (EPA 2012d).

Detailed analytical results for all Phase V Part A surface water samples are provided in the OU3 master project database (see **Appendix A**). The following sections summarize these results.

2.5.2 Asbestos Results

A number of issues were identified with the analytical results that were originally reported by the laboratory. There were several discrepancies in the reported LA concentrations between rapid TAT and the standard TEM analysis and there were unexpectedly high concentrations of LA reported for some locations. In order to investigate these discrepancies, several repreparation analyses were performed (both by the original TEM laboratory and a different TEM laboratory). These analyses confirmed that there had been a misclassification of structures during the rapid TAT analysis and a misidentification of sample IDs during the standard TEM analysis. Because of these issues, it was subsequently determined that the results from the rapid TAT analysis were not usable (EMSL 2013). In addition, results for several standard TEM analyses were also rejected and replaced by the repreparation results. The analytical issues associated with this study are discussed in detail in a memorandum prepared by EPA's Quality Assurance Technical Support (QATS) contractor (CB&I Federal Services [CB&I] 2013a). Potential data quality implications are discussed in Section 12.6.

Table 2-21 summarizes the results[4] of surface water analysis for Part A of the Phase V investigation. As seen, LA was detected in all samples collected in Rainy Creek at LRC-6 and total LA concentrations ranged widely from 10 MFL to 420 MFL, with the highest concentration measured during the first round of sampling (April 25, 2012). Concentrations of LA for structures longer than 10 µm in length ranged up to 66 MFL.

Results from the peristaltic pump pilot study are also included in **Table 2-21**. These results were collected in pilot studies designed to determine if there was a meaningful difference in concentration values obtained between various sampling methods. One method evaluated was collecting water using a peristaltic pump and plastic tubing. The assumption was that this

[4] Results presented in this table have been revised to reflect confirmed and corrected values, per the recommended changes summarized in CB&I (2013a).

method may tend to underestimate true LA concentrations due to binding of LA to the peristaltic tubing. Based on the results of the pilot studies, EPA concluded that, in cases where direct sampling of surface water is not possible, sampling with a steel tube is generally preferred over sampling with a peristaltic pump. This decision was based mainly on the results of Pilot Study 1, which suggest that peristaltic pump samples may tend to be biased low, coupled with a general expectation that binding of clumps to stainless steel tubing is likely to be less than binding of clumps to plastic tubing.

In the Kootenai River upstream of Rainy Creek, low levels of LA were detected in three sampling rounds at UKR-0 (both under high flow and low flow conditions), with total LA concentrations generally less than or equal to about 0.7 MFL. The field sampling team was concerned that samples collected at UKR-0 may have been influenced by Rainy Creek due to a sizable eddy observed during sampling at this location (see Appendix C of the *Phase V Remedial Investigation Field Sampling Summary Report,* MWH 2013). The highest concentrations were measured during the first round of sampling. Concentrations of LA for structures longer than 10 µm in length were usually non-detect, with detected concentrations of 0.05 MFL. LA was not detected at sampling locations further upstream (UKR-1 or UKR-3).

In the Kootenai River downstream of Rainy Creek, LA was detected intermittently, with total LA concentrations generally less than or equal to about 1 MFL. Concentrations of LA for structures longer than 10 µm in length were usually non-detect, with detected concentrations of less than or equal to 0.1 MFL. Although total LA was detected in Transect D and E at KR-14 and Transect D at KR-16 during the high flow sampling events, a spatial patterns of asbestos concentration are not evident since most of the transect sample results are non-detect.

2.5.3 Flow Data

The average flow corresponding to each week of the investigation at CC-2, LRC-2 and LRC-6 is shown on **Figure 2-12** and in **Table 2-22**. As expected flow measurements were higher in the initial weeks of the investigation during spring run-off. **Figure 2-13** displays total LA concentrations for each location along with corresponding flow measurements for LRC-6. As seen, LA concentrations at LRC-6 are higher when flow rates are higher and the highest flow rates were measured in late April. Total LA concentrations at LRC-6 are significantly higher than at any of the Kootenai River locations.

2.6 Phase V, Part B (2012)

Part B of the Phase V investigation focused on providing data to support a baseline ecological risk assessment (BERA) for OU3. Surface water results associated with ecological studies are discussed in Section 9.4 (in-stream fish toxicity studies) and Section 10.6 (amphibian community field study).

3 Sediment

Sediment samples were collected at OU3 as part of the Phase I, Phase II, and Phase V sampling programs. Sediment samples were analyzed for a broad suite of analytes, including LA and non-asbestos chemicals. The following sections summarize the sediment field data for each sampling program conducted from 2007 to 2013. Detailed summaries of results for asbestos and for non-asbestos chemicals in sediment samples are provided in **Appendix B** and **Appendix C,** respectively.

3.1 Phase I (2007)

3.1.1 Sampling Design

The objective of the Phase I sampling program was to collect sediment data to obtain a preliminary characterization of the nature and extent of potential sediment contamination related to historical mining, milling/processing, and mine-waste disposal operations.

Figure 2-1 identifies the locations where sediment samples were collected; these samples were co-located with the surface water samples (a description of these stations is summarized in **Table 2-1**). All sediment samples were collected in October 2007. Samples were analyzed for LA, metals/metalloids, petroleum hydrocarbons, anions, and other sediment quality parameters. A broad suite of additional analyses, including PCBs, pesticides, herbicides, VOCs, SVOCs, and PAHs, were performed for sediment samples collected from TP-TOE2 and LRC-2. As noted previously, these locations were selected because they appear to have the best potential of characterizing releases from the mine.

All sediment sampling was conducted by MWH. Detailed information on the Phase I field sampling effort, including all associated field documentation, is provided in the *Phase I Field Sampling Summary Report* (MWH 2007).

After sediment samples were collected in the field, the samples for asbestos analysis were sent to the CDM Federal Programs (CDM Smith) Close Support Facility (CSF) in Denver, Colorado for preparation. At the CSF, each sediment sample was dried and sieved through a ¼-inch screen. Particles retained on the screen (if any) are referred to as the "coarse" fraction. Particles passing through the screen are referred to as the fine fraction, and this fraction was ground by passing it through a plate grinder. The resulting material was referred to as the "fine ground" fraction. The fine ground fraction was split into four equal aliquots. One aliquot of the fine ground material and the coarse fraction material were then shipped to EMSL-Libby for analysis of LA by polarized light microscopy (PLM).

The coarse fractions were examined using stereomicroscopy, and any particles of asbestos (as confirmed by PLM) were removed and weighed in accordance with Libby-specific standard operating procedure (SOP) SRC-LIBBY-01 (referred to as polarized light microscopy – gravitmetric, "PLM-Grav"). The fine ground aliquots were analyzed using a Libby-specific PLM method using visual area estimation (referred to as polarized light microscopy – visual area

estimation, "PLM-VE"), as detailed in SOP SRC-LIBBY-03. PLM-VE is a semi-quantitative method that utilizes site-specific LA reference materials to allow assignment of fine ground samples into one of four "bins", as follows:

- Bin A (ND): non-detect
- Bin B1 (Trace): detected at levels lower than the 0.2% LA reference material
- Bin B2 (<1%): detected at levels lower than the 1% LA reference material but higher than or equal to the 0.2% LA reference material
- Bin C: LA detected at levels greater than or equal to the 1% LA reference material

Analyses of non-asbestos chemicals in sediment were performed by ELI.

Detailed analytical results for all Phase I sediment samples (asbestos and non-asbestos) are provided in the OU3 project database (see **Appendix A**). The following sections summarize these results.

3.1.2 Non-Asbestos Results

Table 3-1 presents summary statistics on the detection frequency and concentration of non-asbestos analytes detected in sediment samples analyzed as part of the Phase I sampling program. As seen, a number of metals/metalloids and anions were detected in sediment. Significant variability is observed in results for metals/metalloids. Most metals were detected at higher concentrations in samples at seep locations (specifically CCS-8). However lead and manganese were reported at concentrations an order of magnitude higher at tailing impoundment locations than at other locations. As shown, only two chemicals (methyl acetate and pyrene) included in the additional broad spectrum suite conducted for select sediment samples were detected. PCBs, pesticides, herbicides, and other SVOCs were not detected. Petroleum hydrocarbons were detected in upper Rainy Creek, the tailings impoundment, the Fleetwood Creek Pond, and at seep CCS-11. Total extractable hydrocarbons were the most frequently detected and the highest concentration was reported in the tailings impoundment.

3.1.3 Asbestos Results

Table 3-2 summarizes the results of the analysis of sediment for LA. As seen, nearly all (22 out of 24) of the sediment samples collected contained LA. In the fine ground fraction, values ranged from non-detect to 7%. In the coarse fraction, levels generally ranged from non-detect to 0.005%. The highest percentages of LA reported in sediment samples were from seep locations, followed by samples from Carney Creek (CC-1) and the tailings impoundment.

Figure 3-1 shows the spatial pattern of LA in the fine fraction of sediment. As shown, LA was detected in most samples, except those collected in the upper-most reaches of Rainy Creek and Fleetwood Creek. Concentrations of 1% or higher (Bin C) were reported for multiple locations. The highest levels observed were in samples collected from on-site seeps.

3.2 Phase II, Part A (Spring/Summer 2008)

3.2.1 Sampling Design

Data from Phase I sampling program provided information on the concentrations of LA and other non-asbestos chemicals in sediment for a single sampling event (conducted in the fall of 2007). Because concentrations of contaminants in sediment could vary over time, the objective of the Phase II Part A sampling program was to collect additional sediment data in the spring and summer of 2008 to characterize any potential temporal and spatial patterns of site-related contaminants in sediment at OU3.

Sediment sampling in the Rainy Creek watershed was conducted under "Element 1" of the Phase II Part A sampling program (see Section 2.2.1). The purpose of this element was to measure contaminants in sediment at the stations sampled in Phase I to characterize levels during spring and summer flow conditions.

This program differed from Phase I in that the tailings impoundment and each of the ponds (the Mill Pond and the ponds on Carney Creek and Fleetwood Creek) were sampled by collecting a series of grab samples rather than a single composite sample. **Figure 3-2** shows the 17 sediment sampling locations in the tailings impoundment. These sample locations were focused mainly in areas that are always or usually inundated with water, since these areas are most likely to serve as habitat for aquatic receptors. At the three other ponds (the Mill Pond and the ponds on Carney and Fleetwood Creeks), a total of 5 sediment grab samples were collected from each pond, including 3 samples from around the margins of the pond (at least 3 feet in from the edge) and 2 samples from near the center of the pond.

Two rounds of sampling were completed – one in June/July 2008 and one in September 2008. All sediment samples were analyzed for LA, metals/metalloids, petroleum hydrocarbons, anions, total organic carbon (TOC), and other sediment quality parameters. Sediments from lower Rainy Creek (LRC-1 to LRC-6) and the tailings impoundment toe drain (TP-TOE2) were analyzed for PCBs to assess the potential effects of use of oil for dust control along the adjacent road. Sediment collected from TP-TOE2 and LRC-2 was also analyzed for VOCs, SVOCs, and cyanide.

The Phase II Part A sampling program also collected sediment samples in the Kootenai River. In brief, the following samples were collected (see **Figure 3-3** for a map of sampling locations):

- Two grab samples from depositional areas located along the north bank of the Kootenai upstream of the mouth of Rainy Creek.
- Two grab samples from depositional areas located along the north bank of the Kootenai downstream of, but within a distance of 0.5-mile downstream from the mouth of Rainy Creek.
- Two locations from the large gravel bar located in the center of the river about 0.5-mile downstream from the mouth of Rainy Creek. One location was on the highest portion on the gravel bar; the other location was at the downstream point of the gravel bar.

All Kootenai River sediment samples were analyzed for LA.

All sediment sampling was conducted by MWH. Detailed information on the Phase II Part A field sampling effort, including all associated field documentation, is provided in the *Phase II Field Sampling Summary Report* (MWH 2009).

After sediment samples were collected in the field, the samples for asbestos analysis were sent to the CDM CSF in Denver, Colorado for preparation. After preparation, samples were sent to the EMSL laboratory in Westmont[5], New Jersey (EMSL-Westmont) and Hygeia for analysis of LA by PLM-VE (and PLM-Grav, if a coarse fraction was present). Analyses of non-asbestos chemicals in sediment were performed by ELI.

Detailed analytical results for all Phase II Part A sediment samples (asbestos and non-asbestos) are provided in the OU3 project database (see **Appendix A**). The following sections summarize these results.

3.2.2 Non-Asbestos Results

Table 3-3 presents summary statistics on the detection frequency and concentration of non-asbestos analytes detected in sediment samples analyzed as part of the Phase II Part A sampling program. As seen, a number of metals/metalloids, anions, and hydrocarbons were detected in sediment in all areas in both sampling events. There is significant variability in concentrations, although temporal patterns are not obvious.

Total extractable hydrocarbons were the most frequently detected hydrocarbons and were detected in at least one sediment sample from each area, with concentrations ranging from 22 milligrams per kilogram (mg/kg) to 2,360 mg/kg. The most frequently detected carbon ranges were C11 to C22 aromatics and C19 to C36 aliphatics; these carbon ranges were detected in about 90% of the samples. PCBs were not detected in sediment samples from lower Rainy Creek and or the tailings impoundment. Sediment samples collected at TP-TOE2 and LRC-2 were also analyzed for PAHs, PCBs, VOCs, SVOCs, and cyanide. With the exception of PAHs, none of these compounds were detected in sediment. PAHs were detected at LRC-2 in Round 1 and methyl acetate was detected at both locations during Round 2. Detection limits for PAHs vary due to varying moisture content in sediment samples and the reported detections at LRC-2 were below detection limits for most other samples.

3.2.3 Asbestos Results

Table 3-4 summarizes the LA results for sediment samples collected during the Phase II Part A sampling program. LA results for the fine ground fraction of sediment samples ranged from non-detect to 5%, with concentrations of <1% or trace reported in most samples. Maximum LA concentrations were reported in sediment samples collected from seep locations. At locations sampled in upper Rainy Creek, LA concentrations were non-detect or trace for all samples.

[5] The EMSL-Westmont laboratory and its staff moved from Westmont to Cinnaminson, NJ in March 2010.

Table 3-5 summarizes the LA results for sediment samples collected from the Kootenai River. As shown, LA results for the fine ground fraction sediment samples from the downstream Kootenai River stations ranged from non-detect to trace. The sample from the upstream station (UKR-2) was non-detect.

3.3 Phase II, Part C (Fall 2008)

3.3.1 Sampling Design

As noted previously, the Phase II Part C sampling program included the collection of environmental samples at two of the three candidate aquatic reference stations, Noisy Creek (NSY-R1) and a tributary to Bobtail Creek (BTT-R1) (see **Figure 2-9**). In addition, sediment samples were also collected from a subset of stations in Rainy Creek (URC-1A, URC-2, LRC-2, LRC-3, LRC-5, and TP-TOE2), Fleetwood Creek (FC-2), and Carney Creek (CC-2) concomitant with the collection of the aquatic community surveys (see Section 10.3) (see **Figure 2-3**). Sediment samples were collected from each station in October 2008. All sediment sampling was conducted by Parametrix (a contractor to Remedium). Detailed information on the Phase II Part C field sampling effort, including all associated field documentation, is provided in the *Final Data Report for the Autumn 2008 Aquatic Data Collection Program* (Parametrix 2009a).

All sediment samples were analyzed for LA, metals/metalloids, TOC, pH, and total solids. After sediment samples were collected in the field, the samples for asbestos analysis were sent to the CDM CSF in Denver, Colorado for preparation. After preparation, samples were sent to Hygeia for analysis of LA by PLM-VE (and PLM-Grav, if a coarse fraction was present). Analyses of non-asbestos chemicals in sediment were performed by ELI.

Detailed analytical results for all Phase II Part C sediment samples (asbestos and non-asbestos) are provided in the OU3 project database (see **Appendix A**). The following sections summarize these results.

3.3.2 Non-Asbestos Results

Table 3-6 presents summary statistics on the detection frequency and concentration of non-asbestos analytes detected in sediment samples analyzed as part of the Phase II Part C sampling program. As seen, a number of metals/metalloids were detected in sediment. There was significant variability in the results for Rainy Creek, Fleetwood Creek, and Carney Creek; however, concentrations of metals/metalloids in sediments from the aquatic reference stations were generally similar.

3.3.3 Asbestos Results

Table 3-7 summarizes the LA results for sediment samples collected during the Phase II Part C sampling program. As seen, LA levels ranged from non-detect to 5% in the fine ground fraction and from non-detect to 10.6% in the coarse fraction. **Figure 3-4** shows the spatial distribution of LA concentrations observed in the Phase II Part C sampling program. LA was not detected in sediment from the off-site reference locations or at the furthest upstream location in upper

Rainy Creek. Trace amounts of LA were reported in upper Rainy Creek and in Fleetwood Creek. Generally the highest levels observed were in samples collected from Carney Creek.

3.4 Phase V, Part A (2012)

Sediment sampling in Part A of the Phase V investigation focused on characterizing the potential nature and extent of asbestos in sediment at locations frequented by recreational visitors along the Kootenai River and Lake Koocanusa.

3.4.1 Sampling Design

The sediment sampling design conducted as part of the Phase V, Part A sampling program is discussed in detail in the *Final Phase V, Part A: Kootenai River Surface water, Sediment, and Activity-based Sampling SAP/QAPP* (EPA 2012d) and summarized below.

Sediment samples were collected from two locations (KR-20, KR-21) in depositional areas of Kootenai River banks and sand bars (see **Figure 2-11**). Samples were collected in September 2012, under low flow conditions when the most sediment was exposed. Each sediment sampling location was selected because it was representative of areas frequently used by recreational visitors. For location KR-20, which was also sampled as part of a recreational ABS effort (see Section 8.3), sediment sampling was performed prior to the start of each ABS event. Sediment samples were also collected from two locations along the banks of Lake Koocanusa, including the McGillivray Campground (LK-1) and the Lake Koocanusa Marina (LK-2) (see **Figure 2-11**).

Each composite sediment sample was comprised of 30 individual sampling points that were approximately equidistant from each other and representative of each recreational area. This sampling method accounts for spatial variability in LA concentrations and provides an average estimate of the LA concentration across the exposed area. Although visible vermiculite data were collected at the time of sediment sample collection, these data were not collected in accordance with the visible vermiculite SOP identified in the governing SAP/QAPP (SOP CDM-LIBBY-06); therefore, these data are not presented in this report but are available in the field log notes.

Sediment sampling was conducted by MWH. Detailed information on the Phase V, Part A field sampling effort, including all associated field documentation, is provided in the *Phase V Remedial Investigation Field Summary Report (MWH 2013)*.

Sediment samples were analyzed for asbestos (no non-asbestos analyses were performed) and were sent to the Soil Preparation Facility (SPF) located in Troy, Montana for processing in accordance with SOP ISSI-LIBBY-01. [Note: The SPF took over all OU3 soil/sediment sample processing from the CSF in 2009; this was the first set of OU3 investigation samples processed at the SPF.]

The fine ground aliquots for each sediment sample were analyzed by EMSL-Libby using the Libby-specific PLM-VE method (SOP SRC-LIBBY-03). There were no coarse fractions for any of the sediment samples from this study.

Detailed analytical results for all Phase V Part A sediment samples are provided in the OU3 project database (see **Appendix A**). The following section summarizes these results.

3.4.2 Asbestos Results

Table 3-8 presents the results for PLM-VE results for sediment samples collected during the Phase V, Part A investigation. As shown, LA was detected in Kootenai River sediment samples collected from the sandbar above the confluence with Libby Creek (KR-21) and the sandbar below the confluence with Rainy Creek (KR-20); both samples were reported as <1% (Bin B2). LA was not detected in sediment samples collected from either of the Lake Koocanusa recreational areas. No other types of amphibole asbestos or chrysotile were detected in any of the sediment samples.

3.5 Phase V, Part B (2012)

Part B of the Phase V investigation focused on providing data to support a BERA for OU3. Sediment results associated with ecological studies are discussed in Section 9.3 (amphibian laboratory toxicity test), Section 9.4 (in-stream fish toxicity studies), and Section 10.6 (amphibian community field study).

4 Groundwater

Groundwater samples were collected at OU3 as part of the Phase II Part B sampling program. Three rounds of sampling were completed for groundwater, occurring in the summer and fall of 2008, and the spring of 2009. Groundwater samples were analyzed for a broad suite of analytes, including LA and non-asbestos chemicals. The following sections summarize the groundwater data collected at OU3 during these sampling efforts. Detailed summaries of results for asbestos and for non-asbestos chemicals in groundwater samples are provided in **Appendix B** and **Appendix C,** respectively.

4.1 Sampling Design

A site reconnaissance effort was conducted by MWH in the fall of 2007 (during the Phase I sampling program) to identify any groundwater wells at OU3. A total of ten wells were identified within the vicinity of OU3 (see **Figure 4-1**). **Table 4-1** summarizes information for each of these wells. Five of the ten wells identified (wells A, C, D, E, and H), as agreed upon with EPA, were sampled as part of the Phase II Part B sampling program. Groundwater samples were collected from wells A, D, and E in each of three sampling events – July 2008, September 2008, and June 2009. Groundwater samples were collected from Well C in September 2008 and June 2009 and from Well H in July 2008 and June 2009. No sample was collected from Well H in September 2008 (Round 2) because the well was dry. All groundwater samples were analyzed for LA, metals/metalloids, petroleum hydrocarbons, anions and other water quality parameters, gross alpha/gross beta, and cyanide. If the total extractable petroleum hydrocarbon (EPH) concentration exceeded 300 micrograms per liter (µg/L), samples were also analyzed for specific EPH compounds (e.g., C9-C18 aliphatics, C19-C36 aliphatics, and C11-C22 aromatics) and PAHs. At the time of sample collection, field measurements of several water quality metrics, including temperature, pH, specific conductance, DO, ORP, and turbidity, were measured using portable field meters.

All groundwater sampling was conducted by MWH. Detailed information on the Phase II Part B field sampling effort conducted in 2008, including all associated field documentation, is provided in the *Phase II Field Sampling Summary Report* (MWH 2009). (Note: A field sampling summary report for groundwater sampling efforts completed in 2009 has not been prepared.)

After groundwater samples were collected in the field, the samples for asbestos analysis were hand-delivered to EMSL-Libby for filtration. (Note: No treatment of the water was performed prior to the filtration.) The resulting filters were analyzed by EMSL-Libby for total LA by TEM. Filters were prepared and analyzed in basic accordance with ISO 10312:1995(E) (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications.

Analyses of non-asbestos chemicals in groundwater were performed by ELI.

Detailed analytical results for all groundwater samples (asbestos and non-asbestos) and field-collected water quality metrics are provided in the OU3 project database (see **Appendix A**). The following sections summarize these results.

4.2 Non-Asbestos Results

Table 4-2 presents summary statistics on the detection frequency and concentration of non-asbestos analytes detected in groundwater samples collected as part of the Phase II Part B sampling program. As seen, a number of inorganic constituents (metals, anions, and nitrogen compounds) were detected in groundwater in all three sampling rounds.

In general, metals were more frequently detected and at higher concentrations in Well A, a shallow groundwater well located in the Carney Creek drainage. Concentrations of nitrogen compounds varied over three orders of magnitude with the highest concentrations observed in Wells D, E, and H. Gross alpha was detected in 11 out of 13 samples and gross beta was detected in all samples, with the highest levels observed in Well E during Round 3. Petroleum hydrocarbons were detected in all wells except Well C. EPH concentrations varied by two orders of magnitude with the highest EPH concentration reported at Well H. PAHs and EPH specific compounds were not detected in any samples selected for these analyses. Toluene was the only volatile hydrocarbon detected and was detected at a concentration of less than 1 µg/L in Wells D and E in September 2008.

4.3 Asbestos Results

Table 4-3 summarizes the LA groundwater concentrations (based on total LA and LA longer than 10 µm) for each well for each sampling event. Total LA concentrations ranged from non-detect to about 65 MFL and LA concentrations for structures longer than 10 µm ranged from non-detect to about 3 MFL. LA was detected more frequently and at higher concentrations in Well E in most sampling rounds. Concentrations of LA in samples from Wells A and H were lower in the spring compared to the winter.

As noted above, collected groundwater samples were not treated (ozonation/UV) prior to filtration to address potential fiber clumping/wall adherence issues (EPA 2008b). As seen in **Table 4-3**, samples collected in Rounds 1 and 2 were not filtered until 3-5 months after sample collection; thus, asbestos concentrations in these samples are uncertain.

4.4 Field Measurement Results

Table 4-4 summarizes field data measurements collected at groundwater wells in July and September 2008 including: temperature, pH, specific conductance, DO, ORP, turbidity, the volume of water extracted, and the flow rate. Because wells are screened at different depths, field measurements vary from well to well. Of note, turbidity was quite high (greater than 2,000 nephelometric turbidity units [NTU]) in the groundwater sample from Well A collected in July 2008. However, groundwater at Well A is shallow (depth to groundwater was measured at only

3.81 feet below the top of the casing during the July 2008 event) and field samplers noted organic material in this sample. Flow rates that could be determined were low for all wells, ranging from 0.25 gallons per minute (gpm) to 0.75 gpm.

5 Soil and Mine Waste from the Mined Area

Figure 5-1 presents an aerial view of the current condition of the mine site and the main surface features. As shown, the mined area was heavily disturbed by the open-pit mining activities, and some areas remain largely devoid of vegetation. There are a number of areas where mine wastes have been disposed, including waste rock dumps (mainly on the south side of the mine), coarse tailings (mainly to the north of the mine), and fine tailings (placed in the tailings impoundment on the west side of the site). All former buildings and mine works at the site have been demolished and removed.

Soil and mine waste materials from the mined area have been sampled as part of the Phase I program in 2007 and the Amphitheater removal effort in 2012/2013. The Phase I sampling was completed in October 2007; these samples were analyzed for a broad suite of analytes, including LA and non-asbestos chemicals. Soil samples collected as part of the Amphitheater removal were analyzed for LA. The following sections summarize the field data for these samples. Detailed summaries of results for asbestos and for non-asbestos chemicals in soil and mine waste samples are provided in **Appendix B** and **Appendix C,** respectively.

5.1 Phase I (2007)

5.1.1 Sampling Design

The objective of the sampling activities conducted as part of the Phase I program was to collect samples from representative types of waste materials and soils in the mined area in order to identify environmental contaminants associated with mine wastes and develop a list of source areas of potential concern. **Figure 5-2** shows the locations where samples of mine wastes and surface soil were collected. **Table 5-1** summarizes each type of soil and mine waste sample. In brief, samples were collected from:

- waste rock piles;
- cover material;
- coarse tailings disposal area;
- tailings impoundment;
- outcrops; and
- materials used for construction of unpaved sections of Rainy Creek Road.

Samples collected from the impounded tailings (MS-4 and MS-5) and the coarse tailings area (MS-6 to MS-9) were collected as an 8-point transect composite collected from the top 12 inches of material. **Figure 5-3** provides a schematic illustration of the sampling procedure for the transect samples. All other samples were collected as surficial (0-6 inches) grab samples.

All samples were analyzed for LA and metals/metalloids. Mine waste rock, tailings, soil from the former mill area, and roadway materials were also analyzed for petroleum hydrocarbons. The three samples of Rainy Creek roadway materials were analyzed for PCBs (based upon reports that oil had been used in the past to control dust on mine roads and PCB oils were

present at the mine in the past). Samples collected from the fine tailings impoundment were analyzed for a broader suite of potential contaminants, including pesticides, VOCs, SVOCs, PAHs, and cyanide, as well as PCBs, petroleum hydrocarbons, anions, and other soil quality parameters.

All soil and mine waste sampling was conducted by MWH. Detailed information on the Phase I field sampling effort, including all associated field documentation, is provided in the *Phase I Field Sampling Summary Report* (MWH 2007).

After soil and mine waste samples were collected in the field, the samples for asbestos analysis were sent to the CDM Smith CSF in Denver, Colorado for preparation. After preparation, samples were sent to EMSL-Libby and the EMSL laboratory in Westmont[6], New Jersey (EMSL-Westmont) for analysis of LA by PLM-VE (and PLM-Grav, if a coarse fraction was present). Analyses of non-asbestos chemicals in soil and mine waste were performed by ELI.

Detailed analytical results for all Phase I soil and mine waste samples (asbestos and non-asbestos) are provided in the OU3 project database (see **Appendix A**). The following sections summarize these results.

5.1.2 Non-Asbestos Results

Table 5-2 presents summary statistics on the detection frequency and concentration of analytes detected in soil and mine waste samples analyzed as part of the Phase I sampling program. As shown, metals/metalloids were the most frequently detected analytes. For organic chemicals, a variety of PAHs and hydrocarbons were detected in several samples, and pentachlorophenol and methyl acetate were also detected in a few samples. Results for soil and mine wastes samples are summarized below, grouped by media type.

Waste Rock Samples

Twenty-nine waste rock samples were collected and analyzed for metals/metalloids and petroleum hydrocarbons. There is substantial variability in the analytical results for metals. Metals detected in less than 5% of the samples include antimony and mercury; both of these metals were detected only in waste rock samples. Thallium was detected in only one of the waste rock samples. Petroleum hydrocarbons, mostly extractable hydrocarbons, were detected in several waste rock samples. Volatile hydrocarbons (C5 to C8 aliphatics, C9 to C10 aromatics, and toluene) were detected at MS-14. In addition, total purgeable hydrocarbons (TPH) were detected at MS-14, MS-18, and MS-28. PAHs were analyzed for, but not detected, at MS-20.

Roadway Samples

Metals, anions, and petroleum hydrocarbons were detected in the three roadway samples collected (MS-1 to MS-3). Most metals were detected at higher concentrations at MS-2. Petroleum hydrocarbons were detected in all roadway sample locations; the highest

[6] The EMSL-Westmont laboratory and its staff moved from Westmont to Cinnaminson, NJ in March 2010.

concentrations of EPH were also observed at MS-2. PCBs, PAHs, and volatile hydrocarbons were not detected in roadway samples.

Tailings Samples

Several metals and anions were detected in the six tailings samples. Most metals were detected at higher concentrations at MS-5. Thallium was detected in two of the tailings samples. One pesticide, pentachlorophenol, was detected at MS-5. Methyl acetate was the only VOC detected above reporting limits and was detected at MS-4 and MS-5. Several PAHs and EPH compounds were also detected at MS-4 and MS-5, but not at other locations. PCBs were not detected in tailings samples.

5.1.3 Asbestos Results

Table 5-3 summarizes the LA results for soil and mine waste samples collected during the Phase I sampling program. Asbestos levels in mine waste are shown in **Figure 5-4**.

All soil and mine waste samples collected had a coarse (> ¼-inch) fraction, which was analyzed by PLM-Grav. All coarse fractions had detectable levels of LA, with concentrations by PLM-Grav ranging from trace to 0.037%. The highest measured LA values by PLM-Grav were generally in waste rock, with 7 out of the 13 waste rock samples having LA concentrations greater than 0.01%.

PLM-VE analyses of the fine ground fraction showed that LA concentrations in the majority of samples were less than 1%. The highest levels of LA were generally measured in waste rock samples. The maximum level of LA in fine ground material (8%) was observed at outcrop location MS-25. LA concentrations greater than 1% were also measured in cover materials and coarse tailings.

5.2 Amphitheater Removal (2012/2013)

The "Amphitheater" is a portion of OU3 used by EPA for staging soil removed from Operable Unit 4 (OU4) (the residential/commercial areas of Libby) before it is transported to the top of the former mine for disposal. The Amphitheater is located adjacent to the Mill Pond on the west side of Rainy Creek Road (see **Figure 5-1**). While considering various alignments for re-routing Rainy Creek as part of a preliminary evaluation of potential site remediation scenarios, Grace discovered asbestos-containing vermiculite waste south of the Amphitheater in October 2011. Subsequent investigation determined that vermiculite is spread across approximately 5 acres of the Rainy Creek floodplain immediately south of the Amphitheater (**Figure 5-5**). This material is purported by Grace to be sediment dredged from the nearby Mill Pond during operation of the mine and periodically spread out on the area below the current Amphitheater area. Laboratory analysis (by PLM in accordance with NIOSH Method 9002) of three grab samples of this vermiculite waste material (see **Figure 5-5**) showed LA concentrations of 3-4% (MWH 2012). **Figure 5-6** (Panel A) shows a photograph of typical asbestos-containing vermiculate waste.

The nature, thickness, and extent of the vermiculite waste was evaluated during an investigation performed in July 2012. Waste thickness ranges from less than one inch near the margins to more than 3 feet in berms and piles on the area south of Rainy Creek (MWH 2012). Because the vermiculite waste contains LA, it is possible the material may enter Rainy Creek (which bisects the waste-covered area), which may increase LA concentrations in surface water in lower Rainy Creek.

5.2.1 Study Design

In 2012/2013, a removal action was performed at the Amphitheater. The purpose of this removal action was to eliminate or mitigate the ongoing release of asbestos-containing vermiculite waste to lower Rainy Creek. The waste was excavated and transported to the disposal area at the top of the former mine. The area was re-graded and re-vegetated to minimize erosion of residual asbestos-contaminated soil and/or vermiculite waste to Rainy Creek. The estimated bounds of the removal action were determined based on field observation and examination of test pits. Photographs presented in **Figure 5-6** show the Amphitheater at the start of the investigation (Panel B) and in various stages of excavation (Panels C and D).

Post-removal soil sampling was conducted to document the levels of LA that remain in the soil following removal activities. A detailed description of the data quality objectives (DQOs), study design, and sampling methods are provided in Part B of the *Work Plan for Removal of Asbestos-Containing Vermiculite Waste Near the "Amphitheater" at Libby Asbestos Superfund Site OU3* (MWH 2012). This work plan was developed by one of Grace's contractors, MWH. Grace performed the removal action and field sampling activities with support from their subcontractor Chapman Construction, Inc.

All removal activities and soil sampling was conducted by MWH. Fifteen 30-point composite samples were collected from grids approximately 125 feet square in size (about one-third of an acre) across the removal area. Six composite soil samples were collected in November 2012, eight composite samples were collected in June 2013, and one composite soil sample was collected in July 2013. Soil samples were collected, handled, and documented in basic accordance with the procedures specified in OU3-specific SOP No. 1, *Soil Sampling for Non-Volatile Organic Compound Analysis*. The presence or absence of visible vermiculite at each of the 30 sub-sampling locations was recorded.

After soil samples were collected in the field, the samples were sent to the Troy SPF for preparation prior to asbestos analysis. After preparation, samples were sent to EMSL-Libby for analysis of LA by PLM-VE (and PLM-Grav, if a coarse fraction was present).

5.2.2 Asbestos Results

Table 5-4 summarizes the LA results for all post-removal soil samples collected from the Amphitheater removal area. As shown, LA was detected by PLM-VE in the fine fraction of all soil samples with concentrations ranging from trace (Bin B1) up to 2%. LA was also detected by PLM-Grav for all three samples that had a coarse fraction.

6 Soil, Duff Material, and Tree Bark from the Forested Areas

Most of the soil, duff material (i.e., leaf litter, pine needles, organic debris), and tree bark data from the forested area surrounding the mine was collected as part of the Phase I sampling program. All Phase I samples were collected in October 2007 and analyzed for LA. In the fall of 2011, a subset of the forest soil samples collected during Phase I was subsequently analyzed for metals/metalloids. Additional data on LA in duff material and tree bark have also been collected as part of the commercial logging ABS study performed in 2012.

The following sections summarize the field data for these samples. A detailed summary of results for asbestos in soil, duff material, and tree bark, are provided in **Appendix B.** A detailed summary of non-asbestos chemicals in soil is provided in **Appendix C.**

6.1 Phase I (2007)

6.1.1 Sampling Design

The objective of the Phase I forest sampling effort was to determine the potential extent and spatial pattern of releases of airborne asbestos from the mine. To facilitate a spatial pattern analysis, samples were collected along a number of transects that radiated away from the mine, with special emphasis on the predominant downwind direction (northeast). **Figure 6-1** shows the transects and locations that were sampled as part of the Phase I sampling program.

Table 6-1 describes the transects where tree bark, soil, and duff samples were collected. At each location shown in **Figure 6-1**, one Douglas fir tree (at least 8 inches in diameter) was selected for tree bark analysis. In selecting the tree for sampling, trees having rough bark were preferred over trees with smoother bark, since it was expected that rough bark would tend to capture and retain airborne asbestos fibers on the bark surface more efficiently. For each tree, a tree bark sample was collected at a height of about 4-5 feet above ground from the side of the tree facing toward the mine site using a 2-inch diameter hole saw. In addition, for about 10% of the selected trees, an increment boring device was used to collect a core sample for tree-ring analysis to determine the tree age. At each location, one 5-point composite soil sample was collected from approximately equally spaced sub-locations around the perimeter of a circle with a radius of about 5 feet, centered on the tree that was selected for bark analysis. At each soil collection sub-location, the duff material that was overlying the surface soil was also collected to determine if this organic debris layer contained a significant fraction of the historically deposited asbestos fibers.

All forest area sampling was conducted by MWH. Detailed information on the Phase I field sampling effort, including all associated field documentation, is provided in the *Phase I Field Sampling Summary Report* (MWH 2007).

All tree bark and duff samples were sent to EMSL-Libby, EMSL-Westmont, or EMSL-Beltsville for preparation and analysis for LA in accordance with SOP TREE-LIBBY-OU3 and SOP DUFF-LIBBY-OU3, respectively. In brief, samples were dried, ashed, weighed, and hand-mixed. An

aliquot of the resulting ash was treated with acid, suspended in water, and filtered onto a 47-millimeter (mm) mixed cellulose ester filter with 0.4-µm pore size. This filter was prepared and analyzed by TEM in basic accordance with ISO 10312:1995(E) (ISO 1995) with all applicable Libby site-specific laboratory modifications.

Soil samples collected in the field for asbestos analysis were sent to the CDM Smith CSF in Denver, Colorado for preparation. After preparation, samples were sent to EMSL-Libby for analysis of LA by PLM-VE (and PLM-Grav, if a coarse fraction was present). Detailed analytical results for all tree bark, soil, and duff samples are provided in the OU3 project database (see **Appendix A**). Section 6.1.2 summarizes the asbestos results for each media.

Age cores were sent to the Tree-Ring Laboratory at the University of Arizona for the estimation of tree age. Section 6.1.3 summarizes the tree age results.

Supplemental Evaluation of Metals

As noted above, in the fall of 2011, a subset of the forest soil samples collected as part of the Phase I investigation were subsequently analyzed for metals/metalloids. The purpose of this effort was to provide site-specific data on metal concentrations in soils that were thought to be representative of reference conditions (i.e., not impacted by mining activities). A total of 12 samples were selected for metals analysis. Samples were selected from the furthest two sampling locations from the distal ends of each of six transects (see **Figure 6-2**), three downwind transects (circled in white) and three cross-wind/upwind transects (circled in green). All samples were analyzed for metals/metalloids by ELI.

Detailed analytical results for all forest soil samples analyzed for metals/metalloids are provided in the OU3 project database (see Appendix A). Section 6.1.4 summarizes the metals/metalloid results for the forest soil samples.

6.1.2 Asbestos Results

Table 6-2 summarizes the total[7] LA results for each tree bark sample. In this table, results are presented as a surficial loading estimate (i.e., million LA structures per square centimeter of bark surface area [Ms/cm^2]). A map of these results is shown in **Figure 6-3**. Maximum concentrations were observed in the predominant wind direction towards the northeast. A spatial plot of total LA surface loading levels for tree bark as a function of distance from the mine is shown in **Figure 6-4**. Total LA tree bark surface loading levels ranged from non-detect to 16 Ms/cm^2. Generally, total LA levels are highest within about 4 miles of the mine. Total LA levels for tree bark samples collected 4 or more miles from the mine were less than 1 Ms/cm^2. **Figures 6-5 to 6-11** present the tree bark results in a profile view for each transect.

[7] Total: all LA structures observed and recorded during the TEM analysis (i.e., all structures longer than 0.5 µm with an aspect ratio of 3:1 or greater).

Table 6-3 summarizes the LA results for all forest soil samples collected during the Phase I sampling program. **Figure 6-12** shows a map of PLM-VE LA results for forest soil samples. As shown, nearly all forest soil samples had a coarse fraction. Most PLM-VE and PLM-Grav results were non-detect. Trace LA concentrations were observed in 7 samples within 2 miles of the mine, 6 of which were located northeast of the mine. Three samples had LA concentrations above trace concentrations. The maximum LA concentration was reported in SL-135-01, which is located one half mile from the mine across gradient from the primary downwind direction.

Table 6-4 summarizes the total LA results for each duff sample. In this table, results for total LA are presented on a dry weight basis as million structures per gram of duff (Ms/g) and as mass percent (grams of LA per 100 grams of duff material). However, because estimates of mass percent are uncertain as a consequence of the calculation approach, reporting duff concentrations as Ms/g is preferred. **Figure 6-13** shows a map of the total LA results duff samples, expressed as Ms/g. A spatial plot of total LA concentrations in duff as a function of distance from the mine is shown in **Figure 6-14**, expressed as Ms/g. Generally, LA concentrations are higher in duff samples collected within 2 miles of the mine in all directions. Total LA in duff samples ranged in concentration from non-detect to about 3,200 Ms/g, with the majority of sample concentrations falling below 1,000 Ms/g. Total LA concentrations were greater than 1,000 Ms/g in nine samples. **Figures 6-15 to 6-21** present the duff results in a profile view.

Figure 6-22 presents a map of LA results for tree bark, soil, and duff material at each location.

6.1.3 Tree Age Results

Detailed results of the tree age assessment were presented in Sheppard (2007). **Table 6-5** summarizes the estimated tree age for all collected age cores. The twelve trees selected for this analysis ranged in age from 29 to 100 years old (average age was 69 years). The oldest trees sampled were in SL15 (about 5 miles from the Mine, 30° counter clock-wise from the approximate primary downwind direction). In **Figure 6-23**, Panel A presents the tree diameter measured in the field relative to the tree age (as determined by the age cores) and Panel B presents the measured LA surface loading level on the tree bark relative to the tree age. As shown, the age of coniferous trees in this area cannot be accurately predicted based on measured tree diameter. In addition, there does not appear to be a correlation between the age of the tree and the level of LA surface loading measured on the tree bark.

6.1.4 Metals Results

Table 6-6 presents summary statistics for metals for forest soil samples from the downwind transects and the cross-wind/upwind transects. Statistical comparisons of these two datasets were made using the two-sample hypothesis testing approach for datasets with non-detects (Gehan test) provided in ProUCL v4.00.05 (EPA 2010b). There was no statistically significant difference between samples from the downwind transects and the cross-wind/upwind transects. **Table 6-7** presents the summary statistics for metals for all forest soil samples.

6.2 Re-analysis of Phase I Tree Bark and Duff Samples (2013)

6.2.1 Study Design

As described above, extensive data on LA in tree bark and duff were collected in the forested area near the mine site as part of the Phase I investigation for OU3. As part of the Phase I investigation, a single TEM analysis was performed for each tree bark and duff sample. However, in subsequent investigations, tree bark and duff samples were analyzed in triplicate to account for within-sample heterogeneity.

In order to investigate the potential sample heterogeneity in the Phase I tree bark and duff samples, a subset of the Phase I samples were selected for replicate analysis in 2013. Six tree bark and six duff samples, representative of transect locations near (stations SL15-02 and SL45-02), intermediate (stations SL45-07 and SL45-08), and far (stations SL45-15 and SL45-16) from the mine in the prevalent downwind direction, were selected for replicate analysis (**Figure 6-24**). Selected samples were retrieved from archive and two analysis replicates were prepared for each sample by EMSL-Libby, taking additional aliquots of the ashed residue and prepping/analyzing each aliquot in parallel. Samples were analyzed by TEM in basic accordance with ISO 10312:1995(E) (ISO 1995) with all applicable Libby site-specific laboratory modifications. In brief, all structures that were greater than or equal to 0.5 µm in length and had an aspect ratio of at least 3:1 were recorded.

6.2.2 Asbestos Results and Interpretation

Table 6-8 summarizes the total LA results for each replicate for each tree bark sample (Panel A) and duff sample (Panel B); this table also presents the mean LA levels across all three replicate analyses. As shown, mean total LA bark surface loading levels ranged from 0.0034 to 4.4 Ms/cm^2 and mean total LA concentrations in duff ranged from 16 to 1,964 Ms/g. A map of these tree bark and duff results using a color-coding system for value ranges is shown in **Figure 6-24**.

The results for all tree bark and duff replicates were compared using the Poisson ratio comparison test (Nelson 1982). As shown, there were several instances where the original results (i.e., Replicate #1 results from the 2007/2008 analyses) were statistically different from the 2013 replicate results, based on a 90% Poisson confidence interval. However, it is possible that this is due to the limited number of grid openings that were examined during the original analysis. In several cases, only one or two grid openings were examined in the original analysis, which means that these analyses have higher analytical uncertainty. Even when LA levels were statistically different between replicates, results were usually within a factor of 2-3.

6.3 "Near Mine" Commercial Logging Investigation (2012)

The purpose of this investigation was to collect air samples during commercial logging activities to provide measured data on potential exposures to LA for workers involved in commercial logging activities in the forest near the mine. Available data on levels of LA measured in tree bark, soil, and duff indicate that, in general, the levels of LA tend to decrease

with distance away from the center of the mine (see Section 6.1.2). The commercial logging study was performed in an area close to the mine (in the downwind direction), where high concentrations of LA have been reported in tree bark and duff in previous studies. This study area (see red triangle in **Figure 6-25**) was chosen to be representative of the high end of the potential exposures that may occur and is located on Kootenai Development Company property (a restricted area).

A detailed description of the DQOs, study design, and sampling and analysis methods are provided in the *2012 Commercial Logging Activity-Based Sampling SAP/QAPP* (EPA 2012c).

6.3.1 Study Design

As part of this investigation, samples of tree bark and duff material within the study area were collected and analyzed for LA to characterize the level of environmental contamination. Sampling efforts were conducted in the September of 2012. A total of five tree bark and five duff samples were collected from locations that were spatially representative of the study area (see sample collection points shown in **Figure 6-25**). For tree bark, each sample was a composite consisting of five cores, collected by cutting a circle of bark with a hole saw, from five different trees. For duff, each sample was a composite representative of five sub-locations (collocated with the trees sampled for bark).

All tree bark and duff samples were sent to EMSL-Cinnaminson for preparation and analysis for LA in accordance with SOP TREE-LIBBY-OU3 and SOP DUFF-LIBBY-OU3, respectively. In brief, samples were dried, ashed, weighed, and hand-mixed. Samples of bark and duff were first ashed at high temperature to remove organic matter. A portion of the ashed residue was suspended in acid to dissolve non-asbestos mineral salts, and then diluted in water for filtration through a filter.

During the original analysis (performed in 2012), one TEM analysis was performed for each tree bark and duff sample. In 2013, two additional replicate analyses were performed for each tree bark and duff sample collected from the commercial logging area (i.e., two new filters were prepared using a new aliquot of ash and analyzed in parallel).

All bark and duff filters were analyzed for LA using TEM in basic accordance with ISO 10312:1995(E) (ISO 1995) with all applicable Libby site-specific laboratory modifications. In brief, all structures that were greater than or equal to 0.5 µm in length and had an aspect ratio of at least 3:1 were recorded.

6.3.2 Asbestos Results

Table 6-9 summarizes results for the original analyses as well as the results for the two replicate analyses performed in 2013 for tree bark (Panel A) and duff (Panel B). As shown, LA was observed in all five tree bark samples and across all replicates, with concentrations ranging from 0.4 to 12 Ms/cm^2. LA was also observed in all five duff samples and across all replicates, with concentrations ranging from 146 to 637 Ms/g. On average, the total LA levels for tree bark

measured during this commercial logging study (3 Ms/cm^2) were similar to those measured within this area as part of the Phase I study. Average levels in duff measured during this study (385 Ms/g) tended to be somewhat lower than expected compared to the Phase I study, which indicated duff levels higher than 1,000 Ms/g for this area.

7 Ambient Air

Air monitoring under ambient conditions at OU3 was completed as part of the Phase I and Phase II Part B sampling programs. Two rounds of monitoring were performed, the first occurred in the fall of 2007 and the second in the summer/fall of 2008. Ambient air samples were analyzed for LA. The following sections summarize the ambient air field data. A detailed summary of results for asbestos in ambient air is provided in **Appendix B.**

7.1 Phase I (2007)

7.1.1 Sampling Design

The objective of the Phase I sampling program was to collect data to obtain a preliminary characterization of the nature and extent of potential contamination related to historical mining, milling/processing, and mine-waste disposal operations. Because wind speed and direction are variable, eight stationary air monitors were placed in two concentric rings around the mine area to evaluate asbestos concentrations in ambient air at the mine. The first ring was placed close to the boundary of the disturbed mine area, and the second ring was close to the perimeter of the property owned by KDC. **Table 7-1** summarizes ambient air monitoring locations and **Figure 7-1** shows the locations for the ambient air monitors. Each ambient air sample was collected over a period of 5 days. A total of four sampling events were conducted from October 2 to 22, 2007.

All ambient air monitoring was conducted by MWH. Detailed information on the Phase I field sampling effort, including all associated field documentation, is provided in the *Phase I Field Sampling Summary Report* (MWH 2007).

The ambient air filters were sent to EMSL-Westmont for analysis of asbestos by TEM. Filters were prepared and analyzed in basic accordance with ISO 10312:1995(E) with all applicable Libby site-specific laboratory modifications.

Detailed analytical results for all Phase I ambient air samples are provided in the OU3 project database (see **Appendix A**). The following section summarizes these results.

7.1.2 Results

Table 7-2 presents the LA (total and phase contrast microscopy-equivalent [PCME]) air concentrations for all ambient air samples collected as part of the Phase I sampling program. All filters were able to be prepared directly for analysis by TEM. As shown, all samples were non-detect (most samples had an analytical sensitivity of about 0.0005 per cubic centimeter (cc^{-1})).

7.2 Phase II, Part B (2008)

7.2.1 Sampling Design

Although all the Phase I ambient air samples were non-detect, these data were not considered to be sufficient to conclude ambient air was not of concern because they were collected during a

time of frequent rain (so the potential for release may have been reduced) and because they only spanned a time period of 20 days. Thus, additional ambient air data were collected as part of the Phase II Part B sampling program.

A total of eight stationary ambient air monitors were established around the perimeter of the mined area. The locations of these monitoring stations are shown in **Figure 7-2**. In this figure, stations A-4, A-5, A-6 and A-8 were placed at the same locations as were sampled in Phase I, while stations A-9 to A-12 were new stations. As indicated, five stations were located to the north and east of the mined area, since available meteorological data indicate that the predominant wind direction is to the northeast. Three stations were located along the southern perimeter to capture any releases that may occur during wind reversals. Each ambient air sample was collected over a period of 5 days. A total of eight sampling events were conducted from July 7 to October 17, 2008.

All ambient air monitoring was conducted by MWH. Detailed information on the Phase II Part B field sampling effort, including all associated field documentation, is provided in the *Phase II Field Sampling Summary Report* (MWH 2009).

The ambient air filters were hand-delivered to EMSL-Libby for analysis of asbestos by TEM. Filters were prepared and analyzed in basic accordance with ISO 10312:1995(E) with all applicable Libby site-specific laboratory modifications.

Detailed analytical results for all Phase II ambient air samples are provided in the OU3 project database (see **Appendix A**). The following section summarizes these results.

7.2.2 Results

Table 7-3 presents the LA (total and PCME) air concentrations for all ambient air samples collected as part of the Phase II sampling program. All filters were able to be prepared directly for analysis by TEM. As shown, LA was detected in one or more ambient air samples at stations A-5, A-6, A-9, and A-11. Stations A-5, A-6, and A-11 are located northeast of the mine (in the predominant downwind direction). However, the highest concentration of LA in ambient air was reported at station A-9, located south of the mine.

8 Activity-Based Sampling (ABS) Air

ABS is a standard sampling technique that is used to measure air concentrations during disturbances of asbestos-contaminated materials. During ABS, air monitors are worn by personnel that are engaged in a variety of source disturbance activities, and the resulting air filters are analyzed for asbestos to determine the asbestos air concentration. These air concentrations can then be used to estimate exposures for the purposes of evaluating potential human health risks.

ABS air samples have been collected at OU3 as part of several sampling programs to evaluate a variety of source disturbance scenarios. All collected ABS air samples were analyzed for LA. The following sections summarize the ABS air data from these sampling programs. A detailed summary of results for asbestos in ABS samples is provided in **Appendix B.**

8.1 Phase III (2009)

8.1.1 Sampling Design

A range of different human receptors may be exposed to LA in OU3, including trespassers or "rockhounds" in the mined area, recreational visitors in the forested area and along OU3 streams and ponds, as well as wood harvesters, U.S. Forest Service (USFS) workers, and fire fighters in the forested area.

The Phase III sampling program focused on the collection of ABS data to evaluate LA exposures to recreational visitors in the forested area during the following types of activities:

- Walking or hiking in the forest area around the mine site
- Riding an all-terrain vehicle (ATV) in the forest area around the mine site
- Sawing trees or stacking wood with potentially contaminated tree bark
- Actively disturbing soil and duff when clearing a camping area or building a fire
- Inhalation of smoke from burning wood with contaminated tree bark

A total of 20 ABS areas (see **Figure 8-1**) were identified as candidate areas for evaluation in Phase III. These areas were selected based primarily on a consideration of the large-scale spatial variability of measured LA levels in forest soil, duff, and tree bark (see Section 6), as well as inspection of available maps on roads, trails, and terrain in OU3. Eleven of these areas (shaded in yellow in **Figure 8-1**), those that tended to be predominately in the downwind direction (north-northeast of the mine), were selected for ABS evaluation.

For each ABS area, two ABS personnel performed the following scripted activities:

ABS Scenario	Time (minutes) (a)		Person	
	Start	Stop	No. 1	No. 2
ATV riding	0	20	ATV (lead)	ATV (follow)
	20	40	ATV (follow)	ATV (lead)

ABS Scenario	Time (minutes) (a)		Person	
	Start	Stop	No. 1	No. 2
Hiking	0	20	Hike (lead)	Hike (follow)
	20	40	Hike (follow)	Hike (lead)
Campfire Building	40	60	Collect wood for campfire	
	60	70	Dig Fire Pit	
	70	100	Build and stand near campfire (b)	
(a) For some scenarios, the sampling duration was decreased to reduce the potential for filter overloading. (b) For safety reasons, this activity did not occur in the ABS area, but was conducted on Grace-owned property near Rainy Creek Road and Highway 37 (the area formerly known as the Flyway) using the wood collected from the ABS area. This activity lasted from 20 minutes to 30 minutes.				

As shown, a set of two ABS samples were generated for each person for ATV riding and hiking scenarios and three ABS samples were generated for each person for fire building scenarios. ABS events were conducted at each area approximately every 10 days, starting at the end of August through the beginning of November 2009.

All ABS was conducted by MWH. Detailed information on the Phase III field sampling effort, including all associated field documentation, is provided in the *Phase III Activity-Based Sampling Summary Report* (MWH 2010).

The ABS air filters were sent to Hygeia for analysis of LA by TEM. Filters were prepared and analyzed in basic accordance with ISO 10312:1995(E), with all applicable Libby site-specific laboratory modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-00028, LB-000030, LB-000053, LB-000066, and LB-000085.

Detailed analytical results for all Phase III ABS air samples are provided in the OU3 project database (see **Appendix A**). The following section summarizes these results.

8.1.2 Results

Table 8-1 presents the detection frequency and summary statistics for total and PCME LA in ABS air for each activity (ATV riding, hiking, fire building/burning) stratified by ABS area. As shown, 6 to 8 sampling rounds were conducted for each ABS area. All ABS samples were able to be prepared directly, and all samples achieved the target analytical sensitivity of 0.0060 cc^{-1}. Each field sample was evaluated until a minimum of 2 grid openings in each of 2 grids was examined, the target sensitivity was achieved, 50 LA structures were observed, or an area of 0.5 square millimeters (mm^2) was examined (approximately 50 grid openings (GOs)).

The mean concentration of total LA varied over an order of magnitude depending on the ABS area and the activity performed (see **Figure 8-2**). LA was more frequently detected and at higher concentrations for individuals involved in fire building/burning. Clear spatial patterns are not

apparent, but there is a general tendency for air samples from ABS areas located 6-8 miles from the mine to be lower than air samples from ABS areas located closer to the mine.

For the ATV riding scenario, LA was detected most frequently in ABS samples collected in ABS-10, an area located within 2 miles of the mine, where elevated levels of LA in tree bark and duff material were measured in the Phase I investigation. However, detected LA was also reported in some ABS air samples collected in areas farthest from the mine, though at a lower frequency. LA was not detected in ABS samples in areas ABS-01, ABS-02, ABS-05, ABS-06, and ABS-13.

For the hiking scenario, LA was detected most frequently and at higher concentrations in area ABS-13. The frequency of detection tended to be lowest in ABS areas located furthest from the mine. LA was not detected in ABS samples in areas ABS-01 and ABS-08.

For the fire building/burning scenario, LA was detected in one or more samples for all but one ABS area (ABS-14), which happened to be located closest to the mine. In general, the fire building/burning scenario resulted in higher air concentrations than the other two ABS scenarios.

8.2 Phase IV, Part A (2010)

8.2.1 Sampling Design

The Phase IV Part A sampling program focused on the collection of ABS data to evaluate LA exposures to recreational visitors along OU3 streams and ponds, residential wood harvesters, USFS workers, and fire fighters in the forested area (under synthetic fire-fighting conditions). In addition, the Phase IV Part A SAP included a plan for the collection of opportunistic air samples during authentic forest fires in OU3. For the purposes of the Phase IV Part A ABS effort, only a subset of the 11 ABS areas evaluated in the Phase III study were sampled. For most ABS scenarios evaluated in the Phase IV Part A effort, three ABS areas were selected to represent locations "near" (ABS-10), "middle" (ABS-07), and "far" (ABS-02) from the mine (see **Figure 8-3**).

ABS activities were separated into 6 different "scripts" as follows:

> *Script 1.* This script was designed to simulate recreational visitor exposures while hiking along lower Rainy Creek between Highway 37 and the Grace property line (see the "LRC Study Area" in **Figure 8-3**). In this script, two ABS personnel walked up along the banks of the creek, disturbing bushes and other vegetation as needed to move along the bank of the creek. Personnel switched positions (leader/follower) after half of the sampling time has elapsed. A total of 5 sampling events were conducted in August 2010.
>
> *Script 2.* This script was designed to simulate exposures during non-commercial (e.g., residential) wood harvesting activities in the forested area in OU3. The script included

two types of activity – 2A) driving to and from the wood harvesting area, and 2B[8]) felling, limbing, cutting, and stacking harvested wood. Two ABS personnel performed the scripted activities in each ABS area during each sampling event. ABS was conducted in ABS-02, ABS-07, and ABS-10 (see **Figure 8-3**). A total of 5 sampling events were conducted in each ABS area between July and August 2010.

Script 3. The first part of this script (3A, 3B, 3C) was designed to simulate exposures to USFS workers during activities routinely performed as part of the USFS land management responsibilities. The script included three types of activities – 3A) maintenance of roads and trails, 3B) thinning of trees and vegetation, and 3C) surveying trees (i.e., stand examination). The second part of this script (3D, 3E) was designed to simulate exposures to USFS workers during fire-fighting activities. The script included two types of activities – 3D) cutting fire lines by hand using a Pulaski tool, and 3E) cutting fire lines using heavy equipment (e.g., a bulldozer or tractor plow). Two ABS personnel performed the scripted activities in each ABS area during each sampling event. ABS was conducted in ABS-02, ABS-07, and ABS-10 (see **Figure 8-3**). A total of 5 sampling events were conducted in each ABS area between July and August 2010.

Script 4. This script was designed to simulate exposures to ground-based fire fighters from LA in air released by burning of contaminated duff and trees in OU3. Personal and stationary air samples were to be collected during a simulated forest fire, which was to be achieved by the burning two large slash piles in OU3 (see **Figure 8-3** for slash pile locations). However, due to safety concerns, this script was not performed.

Script 5. This script was designed to provide data on exposures to aircraft pilots during fire suppression flights from LA in air released by burning of contaminated duff and trees in OU3. Script 5A was intended to collect data during a simulated forest fire (i.e., the slash pile burn). Script 5B was designed to collect opportunistic samples during authentic forest fires in OU3, by placing an air monitor in the cockpit of responding aircraft. As noted above, the slash pile burn was not conducted and no wildfires have occurred in OU3 since the development of this SAP. Thus, no data have been collected. (Note: Script 5B has been superseded by the *OU3 Wildfire Contingency Air Monitoring Plan* [EPA 2013b].)

Script 6. This script was designed to provide data on residential exposures from LA in air during authentic forest fires in OU3. As noted above, no wildfires have occurred in OU3 since the development of this SAP. Thus, no data have been collected. (Note: This script and the associated SAP Addendum that was created to support a fire fighter ABS effort have been superseded by the *OU3 Wildfire Contingency Air Monitoring Plan* [EPA 2013b].)

[8] After the first round of sampling, this script was split into two parts (2B.1 - felling & limbing activities; 2B.2 - cutting & stacking activities) and, in some cases, script 2B.2 was split across two different filters (filter 'a' and 'b'), to reduce the potential for filter overloading and need for indirect preparation.

All ABS was conducted by MWH. These data are summarized in the *Operable Unit 3 Phase IV Remedial Investigation Field Data Summary Report, Activity–Based Sampling* (MWH 2011).

The ABS air filters were sent to Hygeia for analysis of LA by TEM. Filters were prepared and analyzed in basic accordance with ISO 10312:1995(E) with all applicable Libby site-specific laboratory modifications.

Detailed analytical results for all Phase IV, Part A ABS air samples are provided in the OU3 project database (see **Appendix A**). The following section summarizes these results.

8.2.2 Results

Table 8-2 presents the detection frequency and summary statistics for total and PCME LA in ABS air for each ABS area stratified by script. As shown, there were 10 ABS air samples collected for each script in each ABS area (i.e., 5 sampling events x 2 ABS personnel). Despite attempts to limit particulate loading on the collected air filter (by decreasing the sampling duration, reducing the flow rates, and splitting the sampling across multiple filters) nearly half of all ABS air samples required indirect preparation prior to analysis. Indirect preparation is known to increase structure counts due to dispersion of bundles and clusters (Health Effects Institute – Asbestos Research [HEI-AR] 1991; Breysse 1991). However, for LA, most structures occur as free fibers, and bundles and clusters are not common. Thus, indirect preparation at the Libby site is not believed to be a significant source of bias. Each field sample was evaluated until a minimum of 2 grid openings in each of 2 grids was examined, the target sensitivity was achieved, 50 LA structures were observed, or an area of 1.0 mm^2 was examined (approximately 100 GOs).

The mean concentration of total LA varied over an order of magnitude depending on the ABS area and the activity performed. The frequency of detection and LA air concentrations were generally highest along lower Rainy Creek during simulated recreational activities (Script 1). As shown in **Figure 8-4**, measured air concentrations for several scripts tended to be highest in ABS-07, the "middle" area.

For the residential wood harvesting ABS scenarios (Script 2A and 2B), LA was not detected in any ABS sample from any area for personnel simulating residential wood harvesters driving to and from wood harvest areas (Script 2A). As noted above, Script 2B activities which included cutting and hauling firewood were split into two parts 2.B.1 (felling and limbing) and 2B.2 (cutting and stacking) after the first round of sampling. Of the two scenarios, LA was detected more frequently and at higher concentrations during felling and limbing activities (Script 2B.1). Also, for Scripts 2B.1 and 2B.2, LA was detected more frequently and at higher concentrations in ABS-07. LA was not detected in ABS samples in ABS-02 for Scripts 2A and 2B.

For USFS forest management worker ABS scenarios (Scripts 3A, 3B, and 3C), no single activity was consistency associated with higher LA concentrations than another. However, the frequency of detection and LA concentrations were highest in ABS-07. Also, a higher frequency

of detection was generally observed in all areas for Script 3B (thinning trees). Only one of the 30 ABS air samples collected in ABS-10 had detected levels of LA, and the single detection was associated with Script 3B activities.

For USFS firefighter ABS scenarios (Scripts 3D and 3E), LA concentrations were generally higher than those for forest management activities. The highest level of total LA and PCME LA were observed in ABS-07 for Script 3D (cutting firelines by hand using a Pulaski tool). In addition, the frequency of detection of LA was generally higher in ABS-07 than in ABS-02 or ABS-10.

8.3 Phase V, Part A (2012) ABS

8.3.1 Sampling Design

Part A of the Phase V investigation focused on the collection of ABS data to evaluate exposures to LA by recreational visitors along the Kootenai River. The ABS air sampling was performed on a sand bar in the Kootenai River immediately downstream of the Rainy Creek (station KR-20, see **Figure 2-11**). The ABS sampling design is discussed in detail in the *Final Phase V, Part A: Kootenai River Surface water, Sediment, and Activity-based Sampling SAP/QAPP* (EPA 2012a) and is summarized below.

The ABS script was designed to simulate activities that are representative of actions that might be performed by local river guides and recreational visitors on the sand bar. The ABS script included landing a boat on the sand bar, walking around and simulating an individual fishing along the edges of the sand bar, and departing by boat. A team of two actors landed a boat on the sand bar; the actors shuffled their feet and gently kicked sediment and rock along the edges of the sand bar for five minutes. Then, the two actors walked around the sand bar for 50 minutes, staying near the edge and occasionally crossing through the interior of the ABS area, to simulate an individual moving about the sand bar from one fishing location to another. Once 50 minutes had elapsed, the actors loaded and launched the boat (shuffling their feet and gently kicking sediment and rock in the process for five minutes). The total ABS time interval was 60 minutes.

ABS air samples were collected on the sandbar on the afternoon of September 19, 2012, during low-flow conditions within the Kootenai River. During the ABS event, two replicate ABS air samples were collected for each actor, one using a high volume pump and one using a low volume pump, resulting in a total of four ABS air samples. Only the two high volume filters were analyzed; the two low volume filters were archived. All ABS was conducted by MWH. Detailed information on the Phase V, Part A field sampling effort, including all associated field documentation, is provided in the *Phase V Remedial Investigation Field Summary Report* (MWH 2013).

The ABS air filters were hand-delivered to EMSL-Libby for analysis of LA by TEM. Filters were prepared and analyzed in basic accordance with ISO 10312:1995(E) with all applicable project-

specific laboratory modifications, including the most recent versions of LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085.

Detailed analytical results for all Phase V, Part A ABS air samples are provided in the OU3 project database (see **Appendix A**). The following section summarizes these results.

8.3.2 Results

Table 8-3 presents the results for PCME LA in ABS air for the recreational visitor scenario at the Kootenai River sand bar. Both ABS samples were able to be prepared directly using the high volume filters and both sample achieved an analytical sensitivity of 0.00031 cc^{-1} (better than the required target). Each field sample was evaluated until a minimum of 2 grid openings in each of 2 grids was examined, the target sensitivity was achieved, 25 LA structures were observed, or an area of 20 mm^2 was examined (approximately 2,000 GOs). LA was not detected in either ABS sample.

8.4 "Near Mine" Commercial Logging (2012)

The purpose of this investigation was to collect air samples during commercial logging activities to provide measured data on potential exposures to LA for workers involved in commercial logging activities in the forest near the mine. ABS air sampling was conducted during authentic commercial logging activities in an area near the mine to evaluate potential asbestos exposures.

8.4.1 Study Design

A detailed description of the DQOs, study design, and sampling and analysis methods are provided in the *2012 Commercial Logging Activity-Based Sampling SAP/QAPP* (EPA 2012c). Key elements of the study design and methods are summarized below.

Study Location

Available data on levels of LA measured in tree bark, soil, and duff indicate that, in general, the levels of LA tend to decrease with distance away from the center of the mine (see Section 6.1.2). The commercial logging study was performed in an area close to the mine (in the downwind direction), where high concentrations of LA have been reported in tree bark and duff in previous studies. The study area is shown by the red polygon in **Figure 8-5**. This study area was chosen to be representative of the high end of the potential exposures that may occur and is located on Kootenai Development Company property (a restricted area).

Timing and Duration of the ABS Effort

Commercial logging ABS efforts were conducted in the September of 2012, when environmental conditions were likely to be driest and potential airborne LA releases were highest. There were no *a priori* sampling durations established for this study. Rather, commercial logging workers (contracted by Remedium) were to perform ABS during the logging of approximately 100 trees.

For activities spanning more than two hours, air filters were changed out every two hours to limit potential filter overloading. Although the ABS samples collected in this study were typically 1-2 hours in duration, the concentrations measured are assumed to be representative of exposures that occur over the full course of a work day.

Characterization of Environmental Levels

Samples of tree bark and duff material within the study area were collected and analyzed for LA to characterize the level of environmental contamination; these data are described in Section 6.3.

Characterization of LA Levels in Air During Commercial Logging Activities

ABS samples were collected for a range of activities representative of commercial logging activities including: hand felling, hooking and skidding, mechanical processing, site restoration, and milling processes. These activities are described below and select activities are shown in **Figure 8-6**.

Hand-Felling - The felling of timber is the process of severing the tree from the stump and placing it on the ground. Hand-felling is the traditional method of skilled personnel, herein referred to as a sawyer, utilizing a handheld chain saw to cut the timber. ABS samples for the felling scenario were collected using personal air sampling pumps with the filter located on the shoulder of the sawyer.

Hooking and Skidding - The skidding of timber is the process of dragging felled trees to a centralized location (the landing area) for further processing or transportation. For this study, trees were moved using a cable skidder, which requires an operator to get off the machine to manually attach trees with cables (or chokers). The activity of attaching chokers to logs is commonly referred to as "hooking". ABS samples for this scenario were collected using personal air sampling pumps with the filter located on the shoulder of the hookers/skidders. The samples represent air levels that occurred during both operations.

Mechanical Processing - Timber processing is the act of cutting limbs from the tree and cutting the tree into the desired length and width. Although mechanical processors vary, most utilize an excavator-type machine that mechanically strips limbs from the tree and cuts the tree into desired lengths. Mechanical processors most often have enclosed cabs in which the operator is stationed through the duration of processing activities. ABS samples for this scenario were collected using personal air sampling pumps with the filter located on the shoulder of the operator inside the cab of the mechanical processing machine.

Milling Process – The milling process is the act of removing bark from cut timber and cutting logs to appropriate size and shape for sale. This activity is commonly performed at a mill site that is remote from the forest. However, for this investigation, logs were cut

into slabs and run through a chipper at the on-site landing area to simulate exposures that might occur in an off-site milling operation. ABS samples for this scenario were collected using stationary air monitors located 10 or 30 feet from the chipper.

Site Restoration - Following harvesting operations, site restoration is performed utilizing a bulldozer to remove brush and tree litter from the landing area until the landing area has been cleared and the road restored to its original condition. ABS samples were collected using personal air samplers to represent exposures of the bulldozer operator and also a helper standing on the ground during bulldozing operations.

The ABS air filters were sent[9] to EMSL-Libby for analysis of LA by TEM in basic accordance with Annex E of ISO 10312:1995(E) with all applicable project-specific laboratory modifications, including the most recent versions of LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085. Analysts examined grid openings under low magnification (5,000x) recording only those structures that met PCME counting rules. The target analytical sensitivity for the ABS air samples was 0.0018 cc^{-1}, but the analyst could halt the analysis before achieving the target sensitivity if 25 PCME LA structures were observed, or if the maximum area was examined.

8.4.2 Results

Detailed analytical results for all commercial logging ABS air samples are provided in the OU3 project database (see **Appendix A**).

Table 8-4 summarizes the results that were obtained for each commercial logging ABS air sample. As indicated, a total of 13 air samples were collected, including:

- Three samples associated with timber felling
- Five samples associated with hooking and skidding of felled trees
- One sample associated with on-site mechanical processing of felled trees
- Two samples associated with site restoration activities
- Two samples associated with chipping (a surrogate for off-site milling activities)

The ABS results indicate that highest LA air concentrations occur during activities that cause substantial disturbance of duff and soil (skidding, bulldozing during site restoration activities), while activities that are associated mainly with disturbance of tree bark (sawing, processing, chipping) tend to produce lower LA air concentrations. This observation is consistent with available source media data, which indicate that asbestos levels in duff and soil are substantially higher than in tree bark (see **Table 6-8**). As shown previously (see **Figure 6-20**), highest values for both duff and tree bark tend to occur within a radius of about 2-4 miles from

[9] ABS filters were originally sent to Material Analytical Services, Inc. (MAS) laboratory in Georgia, but were subsequently sent to EMSL-Libby for TEM analysis due to laboratory subcontracting issues.

the mine (this includes the area where the commercial logging study was performed), and values further than about 6-8 miles from the mine tend to be substantially lower.

8.5 2013 ABS Supplemental Analysis

8.5.1 Study Design

Supplemental analysis was performed for selected ABS air samples collected during the Phase III investigation (see Section 8.1) and the Phase IV, Part A investigation (see Section 8.2) (CDM Smith 2013). This supplemental analysis was performed in order to improve (lower) the achieved analytical sensitivity to support risk management decision making with respect to the draft LA-specific toxicity values. Samples were selected from ABS areas representative of three specified distances from the mine, including ABS area ABS-02 (far from the mine, greater than six miles from the mine), ABS-07 (intermediate from the mine, within six miles from the mine), and ABS-10 (near the mine, within 2 miles of the mine). Twelve ABS air samples collected during the evaluation of a recreational hiking scenario in 2009 were selected from the Phase III investigation and sent to EMSL-Libby for supplemental analysis. Twenty-four ABS air samples collected during the evaluation of a residential wood harvester scenario in 2010 were selected from the Phase IV, Part A investigation and sent to the EMSL laboratory in Denver, Colorado (EMSL-Denver) and EPA's Environmental Services Assistance Team Region 8 laboratory in Golden, Colorado (ESATR8) for supplemental analysis. Fifteen ABS air samples collected during the evaluation of a Pulaski digging scenario on 2010 were selected from the Phase IV, Part A investigation and also sent to EMSL-Denver for supplemental analysis.

All filters from the original ABS investigations were retrieved from archive, new grids were prepared, and these grids were analyzed by TEM in basic accordance with ISO 10312:1995(E) (ISO 1995) with all applicable Libby site-specific laboratory modifications using PCME recording rules.

8.5.2 Results

Table 8-5 presents the results of the Phase III supplemental analysis of the hiking scenario ABS samples. The supplemental analyses achieved a sensitivity of 0.00058 cc^{-1} as compared to the sensitivity of 0.006 cc^{-1} achieved in the original analysis. LA was not detected in any sample in the original analysis or in the supplemental analysis.

Table 8-6 presents the results of the Phase IV, Part A supplemental analysis of the residential wood harvesting ABS samples. As shown, a total of 24 samples were analyzed (eight samples from each ABS area). ABS samples included all script activities (i.e., driving to and from the harvest area and cutting and hauling firewood). LA was detected in five ABS air samples; all samples from ABS-02 (far from the mine) were non-detect.

Table 8-7 presents the results of the Phase IV, Part A supplemental analysis of the Pulaski ABS digging samples. Fifteen samples from the original Pulaski ABS digging scenario were selected

for supplemental analysis; however, for three samples, only two additional grid openings were needed to achieve the target supplemental analytical sensitivity, so no additional analysis was performed. None of the ABS air samples collected from ABS-10 (near the mine) had detected LA, but four of the five ABS air samples from ABS-07 (intermediate from the mine) and ABS-02 (far from the mine) had detected LA, with detected PCME LA air concentrations ranging from 0.0061 to 0.049 structures per cubic centimeter (s/cc).

8.6 Souse Gulch Wildfire (2013)

8.6.1 Study Design

Forest fires that occur within contaminated areas of OU3 may result in the release of LA fibers into the air subsequently exposing firefighters and individuals in surrounding downwind areas to LA in air. The USFS Fire Suppression Restriction Zone (FSRZ) shown on **Figure 8-7** is currently defined as the OU3 study area. This is an area inside which the USFS has determined that ground-based firefighters must wear respiratory protection when responding to forest fires.

Based on meteorological data collected at the mine site, the predominant wind direction at OU3 is to the north-northeast (see **Figure 8-8**). Therefore, smoke and LA released from fires in OU3 is most likely to be transported in that direction. It is believed that levels of environmental LA contamination are likely to be highest in areas that are north-northeast of the mine; consequently, sampling air/smoke from fires that occur within several miles of the mine in the north-northeast direction is especially important. Currently the majority of the land north and east of the former mine is owned by the USFS or by logging companies and human occupancy in this area is sparse.

EPA has developed the *Wildfire Contingency Monitoring Plan SAP/QAPP* (EPA 2013b) to monitor air during authentic forest fires in OU3 to determine if disturbance of source materials (i.e., tree bark and duff) due to the wildfire and fire suppression efforts could result in the release of LA into the air. A detailed description of the data quality objectives, study design, and sampling methods are provided in EPA (2013a).

To date, air samples have only been collected as part of one wildfire. Ambient air and ABS samples were collected during the Souse Gulch wildfire that occurred on July 27, 2013. ABS air samples were collected to provide information on the levels of LA in air near ground-based firefighters responding to the wildfire and pilots providing aerial support during fire suppression activities. In addition, ash material from the burn area was collected following the wildfire to provide information on LA levels in ash following a wildfire.

8.6.1.1 Air Sampling

Air samples collected during the Souse Gulch wildfire included ambient air, cockpit air for the responding helicopter, and ABS air near responding ground-based firefighters. All air samples were collected by Grace contractors and hand-delivered to EMSL-Libby for rapid-turnaround

analysis of LA by TEM in basic accordance with ISO 10312:1995(E) (ISO 1995) with all applicable Libby site-specific laboratory modifications. Filters were examined under low magnification (5000x) using PCME recording rules.

Each of these air sample types is described below.

Ambient Air Monitoring

During the Souse Gulch wildfire, ambient air monitoring was conducted at one fixed station and at one mobile station. **Figure 8-7** shows the location of the wildfire and the air monitoring locations.

Based on meteorological data, the stationary air monitoring station (F1) was established west of Lake Koocanusa within the camping area at McGillivray Access. A 24-hour sample was collected from the stationary air monitor on July 27, 2013.

In addition to the stationary monitor, a mobile monitor (M1) was deployed to an area downwind of the fire. The actual location selected for the mobile sampler depended upon the ease of access for the truck hauling the sample equipment and safety concerns for sampling personnel (e.g., conditions of the fire). The monitor was placed on a tripod in the back of the truck. During sample collection, the coordinates of the monitor were recorded, as well as the wind direction and speed. This information was used in combination with data on the fire location, to establish the distance and direction of the monitor relative to the fire. A 4-hour sample was collected from the mobile monitor on July 27, 2013, starting at 10:00 AM.

Aircraft Cockpit Monitoring

Grace's contractor deployed to the Libby Airport to perform activities associated with calibrating and activating the pump to collect cockpit air samples on July 27, 2013, when an aerial response to the Souse Gulch wildfire was deemed necessary by USFS personnel. The air sampling cassette was positioned in the responding helicopter cockpit to represent the pilot's breathing zone. A one-hour sample was collected inside the cockpit on July 27, 2013, starting at 8:51 AM.

Firefighter ABS

When ground-based firefighters responded to the Souse Gulch wildfire, Grace's contractor deployed two individuals with proper health and safety training to the area of the wildfire. Each individual wore a personal air sampler to collect air samples in the immediate vicinity of the USFS firefighting team. There was no established ABS script to be performed; this scenario simply specified that the sampling personnel were to stand near USFS firefighting personnel engaged in fire suppression activities. Each sampling team member donned and activated their personal air pump after arriving at the scene of the fire. Each individual collected four ABS air samples, changing filter cassettes every 30 minutes. A total of 16 ABS air samples were collected

on July 27, 2013, beginning in the early morning (12:50 AM) and ending in the afternoon (2:00 PM).

8.6.1.2 Ash Sampling

Trial burn experiments in wood stoves (Ward *et al.* 2009) and in test burn chambers (EPA 2012b) have shown that the majority of LA fibers are retained in the ash when wood and duff materials are burned under experimental conditions. Thus, it is possible that ash resulting from a wildfire in OU3 could contain concentrated levels of LA and act as a potential source material.

Following the Souse Gulch wildfire, once it was safe to return to the burn area, field personnel collected ash material from the ground surface to provide measured data on the LA levels in ash. There is no existing SOP for the collection of ash material; therefore, ash was collected manually using a trowel, in basic accordance with OU3 SOP No. 1, *Soil Sampling for Non-Volatile Organic Compound Analysis.* In brief, enough ash material was collected from the ground surface (approximately 0-1 inches in depth) to fill a lidded five-gallon container. Material was collected from 30 individual sampling points across the burn area. At the time of collection, no ash aliquots were removed from the bucket for TEM analysis. Rather, the bucket was maintained in archive under chain of custody (COC) by the field contractor. Custody of this bucket was transferred to the Troy SPF on November 5, 2013.

The Troy SPF utilized a riffle splitter to remove three ash samples for TEM analysis[10]. The SPF assigned unique sample identifiers for the three ash samples as follows: SM-20001, SM-20002, and SM-20003. Ash samples were prepared using procedures similar to those specified in Section 6.2 of SOP EPA-LIBBY-2012-11, *Sampling and Analysis of Duff for Asbestos.* In brief, an aliquot of the ash material was acidified, suspended in water, and filtered. A total of three replicate filters were created for each ash sample using additional aliquots of the ash residue; each replicate was analyzed in parallel. All samples were sent to EMSL-Cinnaminson and analyzed by TEM in basic accordance with ISO 10312:1995(E) (ISO 1995) with all applicable Libby site-specific laboratory modifications. All asbestos structures with a length greater than or equal to 0.5 µm and an aspect ratio of at least 3:1 were recorded.

8.6.2 Results

Table 8-8 summarizes results of the air monitoring conducted during the wildfire at Souse Gulch on July 27, 2013. As shown, PCME air concentrations in ambient air measured at the stationary and the mobile monitor stations were non-detect; air concentrations inside the helicopter were also non-detect. PCME LA was detected in two of the firefighter ABS samples; both samples were collected during the initial firefighter response activities. PCME LA air concentrations in ABS air ranged from non-detect to 0.0031 s/cc, with an average concentration

[10] This sampling technique differed from what was originally specified in the sampling plan. This deviation was documented in field modification LFM-OU3-01 (11/6/13) for the *OU3 Wildfire Contingency Monitoring Plan*.

of 0.00031 s/cc. Achieved analytical sensitivities for the ABS air samples ranged from 0.0015 cc^{-1} to 0.0018 cc^{-1}.

Table 8-9 summarizes the total LA air concentrations ash samples collected from the Souse Gulch wildfire burn area. As seen, LA was detected in all three ash samples, with mean total LA concentrations ranging from 44 to 66 Ms/g.

9 Aquatic Toxicity Tests

Ecological risks are usually evaluated using an approach that relies upon multiple lines of evidence. Site-specific toxicity tests are often relied upon to provide information on the response of receptors that are exposed to site media. This may be done either in the field or in the laboratory using media collected on the site. In the toxicity tests, test organisms are exposed to site media and measurements are made of particular endpoints of interest to determine if exposures are having an adverse impact.

At OU3, two toxicity tests were conducted as part of the Phase II sampling program to evaluate the effect of fish and benthic invertebrate exposure to site surface water and sediment, respectively. An amphibian laboratory toxicity test was conducted as part of the Phase V Part B sampling program to determine if exposure of amphibians to LA in sediment from OU3 would result in adverse effects. In addition, in-stream fish studies were conducted as part of the Phase V Part B sampling program to evaluate effects of exposure to fish (trout eggs or fry) to LA in site waters as compared to reference streams. The following sections summarize the study design and results of each toxicity test.

9.1 Fish Surface Water Laboratory Toxicity Test

9.1.1 Test Design

The surface water toxicity test design is detailed in the Phase II Part A SAP (EPA 2008a). In brief, the test was conducted with newly-hatched larval (sac fry) rainbow trout (*Oncorhynchus mykiss*) under static renewal conditions for an exposure duration of 6 weeks. Survival, behavior, and growth were observed during the exposure period, and the histopathology of the fish was examined at the end of the study.

Because the primary focus of this test was on evaluating the potential toxicity of LA in surface water, the water used in the test was selected by monitoring the levels of LA in OU3 waters in 2008, and choosing a time and place that was believed to be near the high-end of the observed range of LA concentrations to collect surface water for use in the toxicity test. Based on a real-time review[11] of the surface water concentrations in samples collected as part of the Phase II Part A sampling program (see Section 2.2), the tailings impoundment (station TP) was selected for evaluation in the site-specific surface water toxicity test. Surface water for use in the toxicity test was collected from the tailings impoundment on May 8, 2008. Water was shipped to Parametrix Environmental Research Laboratory (PERL) (a subcontractor to Remedium) in Albany, Oregon for use in the toxicity tests.

[11] This was accomplished by performing a preliminary rapid turn-around (within 24 hours) TEM analysis of surface water for a subset of the samples collected under Element 2. Rapid turn-around was accomplished by performing the TEM analysis without recording the detailed structure-specific information (i.e., structure type, length, width).

Prior to performing the toxicity tests, a pilot-scale study was conducted to evaluate if the aquaria water circulation system was sufficient to keep LA fibers suspended in the test waters, thus ensuring the homogeneity of exposure solution. As part of this study, triplicate samples were collected from the top and bottom third of the water column in the aquarium and samples were sent to the EMSL Libby laboratory for rapid-turnaround analysis of LA by TEM. The results from this pilot-scale study (see **Table 9-1**) showed that there was no statistically significant difference, based on the Poisson ratio comparison test (Nelson 1982), between water samples collected from the top of the aquarium and the bottom of the aquarium. This indicated that the water circulation system used in the aquaria was effective in ensuring that the LA in the water was well-mixed. Based on these results, the full-scale surface water toxicity test was initiated on May 22, 2008.

The site surface water was used to prepare a series of test dilutions as follows: 100% (undiluted site water), 10%, 1%, 0.1%, 0.01%, 0.001%, 0% (laboratory control water). At the test initiation, samples of the undiluted site surface water were collected and sent to EMSL-Libby for analysis of LA and to ELI for analysis of metals/metalloids. During the larval stage, water was changed once every 10 days, and after swim up once every 3 days, for a total of seven "cycles". For each round of static renewal, one composite water sample of each test dilution was collected shortly after the start of each renewal cycle, and one composite was collected at the end of the cycle. Samples from Cycle #1, Cycle #2, Cycle#4, and Cycle #7 were sent to EMSL-Libby for the analysis of LA by TEM, other water samples were archived at PERL.

9.1.2 Results

Detailed results of the 2008 OU3 site surface water toxicity test are presented in Parametrix (2009b). No significant effects on survival, growth (wet weight, length, condition factor) were detected for any test dilution. In addition, no unique lesions were evident in fish in the LA treatment groups, and the severity of lesions was not related to the LA treatment group.

Table 9-2 summarizes the measured total LA[12] in the site surface water sample collected at the initiation (Day 0) of the study. Based the measured total LA concentration, the water concentrations in each test dilution were expected to be as follows:

Dilution	Expected Total LA Conc. (MFL)*
100%	~30
10%	3
1%	0.3
0.1%	0.03
0.01%	0.003
0.001%	0.0003

*Based on the rapid turn-around analysis results presented in Table 9-2

[12] Total LA: all structures with length greater than 0.5 um and an aspect ratio of 3:1 or greater.

Table 9-3 summarizes the actual measured total LA water concentrations measured in samples from Cycles #1 and #7 for each test dilution. As shown, measured concentrations were significantly lower than expected for these cycles.

Table 9-4 summarizes the actual measured total LA water concentrations measured in samples from Cycles #2 and #4 for each test dilution. As seen, measured concentrations were lower than expected for these cycles.

9.1.3 Fiber Loss Pilot Washing Study

In order to investigate this apparent loss of LA fibers, a pilot-scale washing study was performed. This pilot study was intended to evaluate the hypothesis that LA fibers had adhered to a bio-film that was present in the mixing carboy and aquaria walls.

As described in the Toxicity of Asbestos in Waters from the Libby Superfund Site Operable Unit 3 (OU3) to Rainbow Trout (*Oncorhynchus mykiss*). (Parametrix 2009b**),** in this pilot study, samples collected during Cycles #2 and #4 were analyzed using a 4-step method as follows:

> Step 1: The sample bottle was gently swirled by hand to suspend any loose material and a 40 milliliters (mL) subsample was removed for TEM analysis.
>
> Step 2: A second 40 mL subsample was removed, placed in a clean beaker, and sonicated for 15 minutes. The sample was then analyzed by TEM. The purpose of this sonication was to disrupt and disperse any fibers that were in suspension but clumped together.
>
> Step 3: A solution of 0.1 M sodium chloride + 0.1 M Graham's salt (sodium hexametaphosphate) was added to the sample bottle to restore the sample volume to the original level. The sample bottle was sonicated and treated with UV light and ozone in accordance with Step 6.2 of EPA Method 100.1. The sample was then analyzed by TEM. The purpose of this treatment was to release and oxidize any microbial growth that may have been present on the walls of the bottle that may have trapped fibers.
>
> Step 4: Step 4 was not performed. This step was to determine if any remaining fibers adhered to the bottle wall. Planned steps were to cut the bottle open and remove a piece of the bottle about 1 cm^2 and submit this sample for examination by TEM.

Results from the four samples from Cycles #2 and #4 used in this pilot-scale washing study are shown in **Table 9-5**. Inspection of these findings suggested the following:

- There was a loss of fibers from the water in the sample bottles. This loss could be accounted for by calculating the total amount of LA in the bottles (in the water and on the bottle wall) and dividing by the volume of water in the bottle.
- There was a time-dependent loss of free fibers in the carboy used to hold the site water sample, with the loss beginning to be apparent sometime after the start of Cycle #2 (day 11 of the toxicity test).

- There was a clear loss of fibers in the aquaria during each cycle that cannot be attributed to a loss in the sample bottle.

The reason for the time-dependent loss of fibers in the carboy, the aquaria, and the sample bottles is not certain. However, the release of fibers in the sample bottles by ozonation and sonication suggests that a microbial growth may be occurring that tends to clump fibers together and ultimately binds the fibers to the walls of the container vessel. Thus, trout exposures in the toxicity tests likely diminished substantially as the test progressed and the lack of adverse effects in the study may be due to a lack of exposure and not representative of the true toxicity.

9.2 Aquatic Invertebrate Sediment Laboratory Toxicity Tests

9.2.1 Test Design

The sediment toxicity test design is detailed in the Phase II Part C SAP (EPA 2008c). In brief, site sediments were tested for toxicity using the amphipod *Hyalella azteca* in a 42-day test (EPA 2000; Test Method 100.4) for measuring the effects of sediment associated contaminants on survival, growth, and reproduction. Sediments were also tested for toxicity to the midge *Chironomus tentans* using the life-cycle test (EPA 2000; Test Method 100.5) for measuring effects on survival, growth, and reproduction.

The sediments used in these tests were selected to be near the high-end of the observed range of LA sediment concentrations in site streams and ponds. Based on a review of LA results for sediment samples collected in Phase I and Phase II sampling programs (see Section 3), two on-site locations (CC-1 and TP-TOE2) were selected for evaluation in the site-specific sediment toxicity test. In addition, sediments from the two off-site reference locations (BTT-R1 and NSY-R1) were also evaluated to provide a site-specific frame of reference for interpreting the results. Sediments for use in the toxicity tests were collected from October 14-17, 2008, and shipped to PERL. Aliquots of each sediment sample were also submitted for analysis of LA and metals/metalloids. The sediment toxicity tests were initiated on November 13-14, 2008, for *Hyalella* and *Chironomid*, respectively.

As part of the *Hyalella* toxicity test, a porewater pilot study was also conducted to quantify LA levels within the sediment porewater of each test material at Day 0 (study initiation) and at Day 28 (at the termination of the study portion of the test). Five replicates per treatment were fitted with a suction lysimeter which collected a 20 mL of porewater. Porewater samples from Day 0 were sent to EMSL-Libby and samples from Day 28 were sent to Hygeia for the analysis of LA by TEM.

9.2.2 Results

Detailed results of the 2008 OU3 site sediment toxicity tests are summarized in Parametrix (2009c,d). Neither test organism (*Hyalella* or *Chironomid*) exhibited any statistically significant

difference in survival, growth, or reproduction when compared to both laboratory control sediments and field-collected reference sediments.

Table 3-7 summarizes the measured LA concentrations in the site sediments (toxicity test samples are identified with a '*'). Concentrations of LA were 3% and 5% in the TP-TOE2 and CC-1 sediment samples, respectively, but were non-detect in sediments from the reference areas. **Table 9-6** summarizes measured metal concentrations in site sediments. As seen, concentrations of most metals in sediment were generally higher in samples from OU3 than in samples from reference areas. Also, concentrations of metals in sediment samples from TP-TOE2 were higher than those reported in CC-1.

Table 9-7 summarizes the measured total LA concentrations in the sediment porewater during the *Hyalella* toxicity tests. (Note: In this table, concentrations are expressed as billion fibers per liter [BFL], not MFL.) As shown, porewater concentrations tended to be highly variable across replicates, and concentrations tended to be much higher on Day 0 compared to Day 28. However, these results are likely influenced by difficulties noted in the sample collection process, which resulted in the presence of variable amounts of sediment in the porewater samples.

9.3 Amphibian Sediment Laboratory Toxicity Test

Part B of the Phase V investigation focused on providing data to support a BERA for OU3. The goal of the amphibian laboratory toxicity test was to determine if exposure of amphibians to LA in sediment from OU3 would result in adverse effects on survival, growth, or metamorphosis. Amphibians may be exposed to LA in the aquatic environment both in water and sediment. This investigation focused on the evaluation of LA exposures in sediment because previous attempts at surface water toxicity tests (see Section 9.1) have shown that it is very difficult to maintain exposure conditions for LA in surface water. The following sections discuss the laboratory-based study of effects in developing amphibians exposed to LA in sediment collected from onsite locations.

9.3.1 Test Design

A detailed summary of the DQOs for the amphibian toxicity test are presented in Section 3 of the *Phase V, Part B: 2012 Ecological Investigations SAP/QAPP)* (EPA 2012b), and the detailed field protocol is presented in Appendix A.1 of the SAP/QAPP. Key study design features and the results of the amphibian toxicity test are summarized below.

The assessment endpoints for the amphibian toxicity test were survival, growth, and metamorphosis. Reproduction was considered as a potential endpoint, but the length of time required to assess this endpoint (i.e., 5-6 additional months of exposure), and resources needed to complete a full reproduction study were determined to be impractical to implement. Potential effects on presumptive gonad tissue were proposed as an indirect way to evaluate the reproductive endpoint.

The toxicity test design and results are provided in detail in the *Amphibian Complete Metamorphosis Exposure Study* (Golder Associates, Inc. [Golder] 2013a); a summary of this study is presented below.

Characterization of the Test Sediment

Because the goal was to expose organisms to the maximum sediment concentrations of LA at the Site, prior to conducting the toxicity test, sediment was collected from the tailings impoundment (TP-TOE2) and Carney Creek (CC-1) (see **Figure 3-1**; these two locations have historically and consistently had some of the highest measured LA concentrations in sediment at the mine). For each station, three "lots" of sediment were collected. Five replicate samples of each lot were prepared (dried, sieved, ground) at the Troy SPF and analyzed by PLM-VE at EMSL-Libby. **Table 9-8** (Panel A) presents the results for each replicate. As shown, CC-1 Lot 3 had measured LA levels that were consistently about 7% LA; thus, this sediment lot was selected for use in the toxicity test.

The LA concentration in the OU3 treatment sediment was also measured at the conclusion of toxicity test to verify exposure concentrations. At exposure termination, sediment was collected from each replicate tank, water decanted, and the sediment was prepared at the Troy SPF and analyzed by PLM-VE at EMSL-Libby. **Table 9-8** (Panel B) presents the PLM-VE results measured at the test termination.

In order to determine the potential presence of other contaminants in the TP-TOE2 or CC-1 sediment that could potentially affect the study, an additional aliquot of the sediment sample collected from each station was submitted to ELI for analysis of non-asbestos chemicals prior to initiation of the bioassay. Sediment each site location was analyzed for organochlorine pesticides, chlorinated herbicides, TOC, diesel and gasoline range organics, mercury, total metals, pH, PCBs (as Aroclors), SVOCs, acid volatile sulfide (AVS), and ammonia (as N). **Table 9-9** presents statistical summaries of the non-asbestos results

Toxicity Test Study Design

Amphibian toxicity testing was performed from September 25 to December 28, 2012 at Fort Environmental Laboratories, Inc. (FEL) in Stillwater, Oklahoma. The test organism was commercial field-collected *Rana sphenocephala* (southern leopard frog) larvae obtained from The Sullivan Company in Nashville, Tennessee. Twenty randomly selected Gosner stage 20 larvae from the same clutch were assigned to one of three treatments:

1) laboratory dilution water and inert sterilized sand (control),

2) laboratory dilution water and reference sediment (from a pond in Oklahoma), and

3) laboratory dilution water and field-collected sediment from OU3 containing LA (from CC-1 Lot 3)

Test chambers were 2.5 gallon glass aquaria (tanks) containing 1.5 kilogram (kg) sediment and 6 liters of laboratory dilution water. Exposure was maintained using a flow-through system in which culture water, but not sediment, flowed through tanks at a rate 2.9 volume exchanges per day. Culture water consisted of de-chlorinated (charcoal filtered) tap water.

Each exposure treatment and control/reference was evaluated in quadruplicate (i.e., four replicates), with 20 organisms per replicate. Once all larvae were assigned to an exposure system, daily observations were recorded, including observations of mortality (survival counts), food consumption, developmental stage and metamorph counts, and other observations on occurrence of malformations or other abnormalities (e.g., abnormal swimming behavior, lethargy, loss of equilibrium, malformations, or lesions).

Fluorescent lighting was used to provide a photoperiod of 12 hours of light per day. Tadpoles were fed boiled organic romaine lettuce leaves *ad libitum* (i.e., quantity and frequency of consumption being the free choice of the tadpole). Documentation of the amount of lettuce consumed by the test specimens was recorded by weighing the lettuce after boiling before introduction to the tank and recording weight after removal of any intact waste lettuce. Daily cleaning of the tanks was performed using a turkey baster to remove organism detritus and excrement.

Any dead larvae were immediately removed and preserved, and then necropsied. During the exposure phase, the number of organisms metamorphosed were recorded, as was the time to metamorphosis (TTM) for each larvae, the weight of each newly metamorphosed larvae, and the median time to metamorphosis (MMT) (determined when 50% of the larvae in a given replicate metamorphosed). The exposure phase was terminated when each of the surviving control (treatment #1) larvae completed metamorphosis. Upon exposure termination, test organisms were anesthetized and digital photos were taken to measure snout-vent length (SVL), whole body weight was measured, external malformation was assessed, and blood (plasma) was collected for possible future analysis of thyroid hormone. The test organisms were then euthanized and visceral (internal) abnormalities (if any) assessed in the completely metamorphosed specimens. The head and carcass (with gonads) were preserved and stored at FEL for possible future histopathology.

Water quality characteristics of the laboratory water were monitored bimonthly for pH, DO, conductivity, hardness, alkalinity, ammonia, residual oxidants; and for iodide (I-), PAHs, pesticides, and metals. The laboratory water was analyzed for pesticides, PAHs, and metals on January 28, 2012 and all water quality measurements met EPA's criteria for aquatic toxicity test culture/dilution water. Both the reference sediment and control sediment (sand) were analyzed for chemicals (total metals, PAHs, and organochlorine pesticides/ PCBs) prior to use in the study.

9.3.2 Results

A detailed toxicity test summary report is provided in the *Amphibian Complete Metamorphosis Exposure Study* (Golder 2013a). Results are summarized briefly below.

Assessment Endpoint Results

Survival. **Table 9-10** summarizes survival endpoint statistics for the three treatments: 1) control sediment (sand), 2) reference sediment, and 3) OU3 sediment. **Figure 9-1** presents a graphical illustration of the survival data. As shown, survival at study termination was 81.3% for the control sediment, 61.3% for the reference sediment, and 70.0% for the OU3 sediment. The toxicity test found that mortality in the LA sediment was not significantly different from the control. Although mortality rates above 20% may suggest that test conditions were not adequate, the report concluded that test conditions were not a factor for this study.

Growth. As seen in **Table 9-10**, mean whole body weight and mean SVL of organisms exposed OU3 sediment were statistically different (higher) than the control sediment and the reference sediment.

Metamorphosis. The criterion for test termination was set at 100% completion of metamorphosis in the control group. The percent metamorphic completion in the control and reference sediments was 100%, whereas 40.4% metamorphic completion was observed in the LA sediment treatment. As shown in **Figure 9-2**, Gosner stage development of the three treatment groups was synchronous up until the final stages, and then some differences appeared.

Abnormalities and Malformations. During the course of the study, no signs of overt toxicity or abnormal swimming behavior were noted. In addition, no signs of asynchronous development, malformations or internal abnormalities were observed in organisms from the control, reference, or OU3 sediment treatments. No internal abnormalities were observed during the study or during the necropsy at the conclusion of the study, suggesting that further histological examination of presumption gonad tissue was not necessary.

Exposure Characterization Results

As shown in **Table 9-8** (Panel A), LA concentrations in the OU3 sediment from CC-1 ranged from 4% to 7% LA at the study initiation. At the study conclusion, measured concentrations in the test sediment ranged from 2% to 3% LA (**Table 9-8**, Panel B). This suggests that some of the LA was lost during the study. However, this apparent difference might be due to variability in the analytical measurements rather than to authentic depletion of the test sediment. LA was not detected in either the control sediment or the reference sediment.

Various metals were detected in site sediments (**Table 9-9**); PAHs, PCBs, and pesticides were not detected above reporting limits. Chemicals detected in site sediments are not expected to affect results of the study.

9.4 In-Stream Fish Toxicity Studies

The goal of this study was to expose fish (trout eggs or fry) to LA in site waters to determine if the exposure resulted in an unacceptable ecological risk as compared to that observed in reference streams. A detailed summary of the DQOs for the in-stream fish studies are presented in Section 5 of the *Phase V, Part B: 2012 Ecological Investigations SAP/QAPP* (EPA 2012b), and the detailed field protocol is presented in Appendix A.3 of the SAP/QAPP. Key study design features and the results of the 2012 eyed egg trout toxicity test (Section 9.4.1) and the juvenile trout toxicity test (Section 9.4.2) are summarized below.

9.4.1 2012 Eyed Egg Toxicity Test

9.4.1.1 Study Design

In this study, trout eggs were placed in streambed gravel in both onsite and reference streams to determine if there was a significant difference in hatching success or alevin survival.

Exposure Method

Eyed eggs from native cutthroat trout were obtained from the Montana Fish Wildlife and Parks fish hatchery in Helena, Montana. Eggs were placed in Whitlock-Vibert boxes (30 eggs per box). Whitlock-Vibert boxes contain small chambers in the upper portion of the box to house the eggs (**Figure 9-3**, Panel A). After the eggs hatch and after some of the yolk sac has been absorbed, the larval fish fall from the upper egg chamber into a lower protected "nursery" chamber (**Figure 9-3**, Panel B) where they rest on the bottom until they develop to the swim-up stage (yolk fully resorbed). Each box was enclosed in rigid plastic mesh (3 mm x 3 mm grid size) in order to minimize the escape of the swim-ups and to provide protection from predators (**Figure 9-4**, Panel A). After fitting the mesh, each box was placed into a steel cage filled with coarse gravel, and was equipped with a sampling tube so water could be withdrawn from the box while still in the gravel (**Figure 9-4**, Panel B).

Exposure Locations

A total of six Whitlock-Vibert boxes were placed in lower Rainy Creek, two boxes each at stations LRC-2, LRC-5, and LRC-6 (see **Figure 2-1**). Likewise, a total of six boxes were placed into the gravel of reference streams, three boxes each at upper Rainy Creek station URC-2 and in Noisy Creek (NSY) (see **Figure 2-1**). The creek locations for Whitlock-Vibert box deployment were selected to approximate a natural redd that fish could use for spawning. Typically, such areas had gravel or cobble substrates and were outside locations with high stream velocity. Sites were prepared by raking out a depression in the selected deployment location. In some cases, structures such as boulders, rocks, or logs were placed upstream to create a breakwater area for placement that ensures flow velocities were not excessive. Boxes were placed in the streambed depression oriented perpendicular to creek flow, and then covered with gravel (see **Figure 9-4**, Panel C).

Developmental Controls

Three replicates of 30 eggs each were kept in a spring water-filled plastic containers in the refrigerator at the off-site laboratory. One of the three containers was taken to the field and then returned to represent a field control (designated FC), while the other two were simply maintained in the laboratory as offsite laboratory references (designated R1 and R2). These eggs were identified as "developmental controls", and were used to assess overall egg quality and batch development. Developmental control eggs were monitored twice weekly and the water changed (70% renewal) with fresh, store bought spring water. Temperature in the refrigerator was monitored and adjusted weekly to reflect creek temperature measurements recorded from the warmest study creek.

Timing and Duration of Exposure

Empty Whitlock-Vibert boxes were placed into the streams on May 2, 2012, to allow the gravel deposits in the exposure areas to become equilibrated with the stream. Eyed eggs were added to the boxes on May 8, 2012. The boxes were left in place until all of the viable eggs had hatched and living fry had reached the swim-up stage. For boxes placed in lower Rainy Creek, hatching occurred within about two weeks, and most surviving alevins had reached swim-up by June 8, 2012 (the date of study termination in LRC). For the reference locations, development was slower, requiring about 3-4 weeks for hatching and seven weeks to reach swim-up, with boxes being removed on June 22, 2012. This difference in development rate is attributed to differences in water temperature (see below).

Field Observations

Each box in LRC, URC, and NSY was observed once per week until study termination. During each examination, the number of dead eggs and alevins was recorded. Dead organisms were removed after each observation. General condition and developmental stage of the organisms was recorded, along with any observations of unusual behavior. In addition, water temperature and oxygen saturation level were measured and recorded.

Laboratory Swimming Observations

At the end of exposure, the boxes were removed from the streambed and transported in site water to an onsite laboratory where all remaining living alevins were transferred into aquaria. After a brief acclimation period, the swimming behavior of the alevins was observed for 30 minutes. Then, the fish were sacrificed and the weight and length of each fish was recorded. Each fish was then placed in preservative for transport to a histological laboratory for external examination.

9.4.1.2 Exposure Conditions

Flow. The eyed egg toxicity test was performed during the time period when high flows associated with spring runoff from snowmelt were expected. **Figure 9-5** shows flow data collected at LRC-2 and LRC-6. As seen, in 2012 (black lines), flow at both stations tended to peak in late April, which was several weeks earlier than in previous years.

Temperature. Surface water temperature was monitored continuously at each exposure location using a data logger. Surface water temperature data are shown in **Figure 9-6**. As seen, temperatures at all stations showed a clear diurnal cycle, with average temperatures trending upward by about 1-2 degrees over the duration of the study. Temperatures were generally similar at the three LRC stations, and tended to be about 5 degrees warmer than at the reference stations.

LA Concentrations. Eggs and pre-swim-up alevins reside in the stream gravel, so the exposure medium of chief concern for LA exposure is the gravel pore water. For the boxes in LRC, pore water samples from within the Whitlock-Vibert boxes were collected twice per week. In addition, on May 10th and May 17th, samples of pore water were also collected from the gravel outside the boxes and from the overlying surface water. For the boxes in the reference locations (UCR and NSY), water samples from within the Whitlock-Vibert boxes were collected once per week from one box (selected at random) at each station. All water samples from site and reference locations were analyzed for total LA by TEM by EMSL-Libby or EMSL-Cinnaminson, treating the water with ozone/ultraviolet prior to analysis to remove any biological material that might cause fiber clumping.

Table 9-11 present the water concentrations[13] of total LA and LA structures longer than 10 µm measured as part of the 2012 eyed egg study. **Figure 9-7** presents the water concentration data for May 10 and May 17, 2012, when co-located overlying surface water, pore water inside the Whitlock-Vibert box, and gravel pore water (outside the box) were measured. As shown, the concentrations of LA within the Whitlock-Vibert boxes and the gravel pore water tended to be generally similar, and both were substantially higher than in the overlying surface water. Concentrations of LA were higher at the three LRC stations than in the reference stations, with maximum total LA concentrations greater than 100 MFL. Concentrations tended to be highest in early May and decreased over time as flow decreased.

9.4.1.3 Results

Eyed egg toxicity test results were presented and evaluated in two technical reports (SRC, Inc. [SRC] 2013; Golder 2013b). All data on the occurrence of dead and living organisms recorded during the study were jointly reviewed by Golder and EPA. This was necessary due to

[13] Note: It was determined that some of the water concentrations originally reported by the laboratory were in error. Concentration data presented in Table 9-13 and Figure 9-8 reflect corrected results. See Section 12.6 for additional information.

limitations in the recorded data and discrepancies in the field documentation. **Table 9-12** and **Table 9-13** summarize the data that were agreed upon for the field exposure boxes and the developmental controls, respectively. Values that are based on professional judgment are shown in yellow (**Table 9-13**).

A summary of the egg hatching success and alevin survival rates, as well as a statistical comparison of rates between field and reference locations is presented in SRC (2013). However, interpretation of the results from this study was limited by: 1) the number of organisms that went missing (presumably due to escape, predation, and/or death and decomposition) over the course of the study and 2) the low survival rates in the reference locations and developmental controls. Because of these limitations, the OU3 Biological Technical Advisory Group (BTAG) determined that it was necessary to repeat the in-stream eyed egg fish study in 2013, modifying the study design and protocol to better address the issues that occurred during the 2012 study (EPA 2013a). Results from the 2013 eyed egg fish study are presented below.

9.4.2 2013 Repeat of Eyed Egg Toxicity Test

9.4.2.1 Study Design

The repeat of the cutthroat eyed egg in-stream toxicity test was conducted in accordance with EPA (2012c), as modified by the *SAP/QAPP Addendum: Part B: 2012 Ecological Investigations SAP/QAPP* (EPA 2013a). A detailed description of the data quality objectives, study design, and sampling methods can be found in these documents. Results of the 2013 eyed egg in-stream toxicity test are summarized in the *Data Report: 2013 In Situ Westslope Cutthroat Trout Toxicity Study* (Golder 2014). Key study design features and results are summarized below.

Exposure Method

Eyed eggs from native cutthroat trout were obtained from the Montana Fish Wildlife and Parks (MFWP) Washoe Park Trout Hatchery in Anaconda, Montana on May 4, 2013. Eggs were placed in Whitlock-Vibert boxes (WVBs) (30 eggs per box) later that same day. WVBs are described in Section 9.4.1.1 and shown in **Figure 9-3**. In the 2013 study, the WVBs were modified to reduce the mesh size of the plastic attached to the WVB walls to 100 openings per square inch and remove the sampling ports used to collect pore water from WVBs that contained organisms. These modifications were expected to reduce the chances of alevin escape, as well as provide better protection from predator entry into the boxes.

Exposure Locations

Similar to the 2012, study trout eggs were placed in streambed gravel in both onsite and reference streams to determine if there was a significant difference in hatching success or alevin survival. A total of six WVBs were placed in LRC, two boxes each at stations LRC-2, LRC-4, and LRC-5 (see **Figure 9-8**). Likewise, a total of six boxes were placed into the gravel of reference streams, three boxes each at URC station URC-2 (see **Figure 9-8**) and in NSY (see **Figure 9-9**). The creek locations for WVB deployment were selected to approximate a natural redd that fish

could use for spawning. Typically, such areas had gravel or cobble substrates and were outside locations with high stream velocity. Sites were prepared by raking out a depression in the selected deployment location. In some cases, boulders, rocks, or logs were placed upstream to create a breakwater area for placement to ensure flow velocities were not excessive. Boxes were placed in the streambed depression oriented perpendicular to creek flow, and then covered with gravel.

Developmental Controls

In the 2012 study, three replicates of 30 eggs were kept in spring water-filled plastic containers in a refrigerator at an off-site laboratory to serve as "developmental controls" for the study. The overall survival in the developmental controls was less than 35%, which was much lower than expected if they had been raised in the MFWP hatchery. Therefore conditions for control populations were modified for the 2013 study.

In the 2013 study, two types of controls were evaluated. The first control group was kept at the MFWP Washoe Park Trout Hatchery in Anaconda, Montana. MFWP took a portion of the eggs from the batch utilized in the study and maintained these organisms at the hatchery for the duration of the study. The hatchery measured and reported on egg hatch success, alevin survival, and overall survival success to the time of swim-up.

The second control was a laboratory control maintained at the off-site laboratory at conditions designed to mimic those in the field. Three replicates of 30 eggs were placed inside three WVBs and were kept in individual aquaria (one aquarium per box) filled with water provided by a local hatchery. One of the three groups of 30 eggs were taken into the field and then returned, while the other two were simply maintained in the laboratory. These aquaria were kept in a refrigerator and temperature was maintained using an automated temperature controller to reflect creek temperature measurements recorded from the warmest study creek. Each aquarium was fitted with a filtration/circulation device to provide circulation of the water and to help ensure adequate DO and water movement, which retards fungal growth. DO levels were monitored to ensure optimum DO conditions. Any bubblers used as part of the filtration/circulation system were located such that the bubbles did not come in contact with either eggs or alevins. Water in each aquaria was changed (70% renewal) twice weekly with hatchery water.

Organisms in the laboratory controls were monitored twice weekly using similar handling procedures as implemented in the field (i.e., organisms were counted, any dead organisms were removed, surviving organisms were transferred to a new WVB). The rate of hatching and development in the laboratory controls was used as an indicator of expected developmental rate in the field boxes, but actual time to reach swim-up was to be determined by direct observations of the organisms in the field.

Timing and Duration of Exposure

Empty WVBs were placed into the streams on April 20, 2013, to allow the gravel deposits in the exposure areas to become equilibrated with the stream. Eyed eggs were added to the boxes on May 4, 2013. The boxes were left in place until all of the viable eggs had hatched and living fry had reached the swim-up stage. For boxes placed in LRC, hatching occurred within about two weeks, and most surviving alevins had reached swim-up by May 30, 2013 (the date of study termination in LRC). For the reference locations, development was slower, requiring about 3-4 weeks for hatching and seven to eight weeks to reach swim-up, with boxes being removed on June 19, 2013. This difference in development rate is attributed to differences in water temperature (see below).

Field Observations

Each box in LRC, URC, and NSY was observed twice per week until study termination. This observation period was more frequent than occurred during the 2012 study, to limit the potential that dead organisms could disintegrate or that mold could infect other organisms between observation periods. During each examination, the number of dead eggs and alevins were recorded. Dead organisms were removed after each observation. General condition and developmental stage of the organisms was recorded, along with any observations of unusual behavior. In addition, water temperature and oxygen saturation level were measured and recorded.

Laboratory Swimming Observations

At the end of exposure, the boxes were removed from the streambed and transported in site water to an onsite laboratory where all remaining living alevins were transferred into aquaria. After a brief acclimation period, the swimming behavior of the alevins was observed for 30 minutes. Then, the fish were sacrificed and the weight and length of each fish was recorded. Each fish was placed in preservative for transport to a histological laboratory for external examination.

Exposure Characterization

Eggs and pre-swim-up alevins reside in the stream gravel, so the exposure medium of chief concern for LA exposure is the gravel pore water. Pore water was collected from within a "dummy" WVB (a box placed within the sediment without any organisms added) using a sampling port. In addition, overlying surface water was also collected near the WVBs. For boxes in LRC, pore water and surface water samples were collected twice a week at each station. For boxes in the reference locations (UCR and NSY), surface water and pore water samples were collected once a week at each station.

All surface water and pore water samples from site and reference locations were analyzed by EMSL-Libby or EMSL-Cinnaminson for total LA by TEM, treating the water with

ozone/ultraviolet prior to analysis to remove any biological material that might cause fiber clumping.

9.4.2.2 Exposure Conditions

Flow. The eyed egg toxicity test was performed during the time period when high flows associated with spring runoff from snowmelt were expected. **Figure 9-10** shows flow data collected at LRC-2. As seen, flow at the station began to peak in late April, with high flows continuing throughout May.

Temperature. Surface water temperature was monitored continuously at each exposure location using a data logger. Surface water temperature data are shown in **Figure 9-11**. As seen, temperatures at all stations showed a clear diurnal cycle, with average temperatures trending upward by about 1-2 degrees over the duration of the study. Temperatures were generally similar at the three LRC stations, and tended to be about 5-10 degrees warmer than at the reference stations.

LA Concentrations. **Table 9-14** present the LA concentrations in water collected during the 2013 eyed egg study; concentrations are presented as total LA and LA > 10 µm. For pore water, total LA water concentrations ranged from 8 to 70 MFL for LRC stations, with the highest concentrations reported for station LRC-4 at the beginning of the study. For surface water, total LA concentrations ranged from 0.12 to 42 MFL, with the highest concentrations generally observed at station LRC-4.

Figure 9-12 presents a graphical illustration of the surface water and pore water concentrations for total LA at the LRC stations. As shown, the concentrations of LA within the gravel pore water tended to be higher than in the overlying surface water. Concentrations tended to be highest in early May and decreased over time. The highest concentrations tended to coincide with peak flows (see **Figure 9-10**).

9.4.2.3 Results

A detailed summary of the egg hatching success and alevin survival rates, as well as a statistical comparison of rates between field, reference locations, and negative controls is presented in Golder (2014). The interpretation of these toxicity test results is reserved for the ecological risk assessment.

Hatching Success and Survival Rates. **Table 9-15** presents a summary of the final organism counts for each WVB box and each station. **Table 9-16** presents hatching success rates, alevin survival rates, and overall survival rates for each box, station, and reach. As shown, hatching success rates ranged from 57% to 75% in LRC and from 73% to 76% in the reference locations. The hatching success rate for the eyed eggs maintained at the hatchery was 77 out of 140 (55%), which was significantly lower than the hatching success rate at the reference locations and in the negative controls (74%). The reason for the lower hatching success in the hatchery control is

not known. Alevin survival ranged from 82% to 95% in LRC and from 85% to 96% in the reference locations. Survival of hatchery alevins (88%) was generally similar to rates in the field.

Swimming Behavior. A higher occurrence of observed swimming abnormalities was observed in the Noisy Creek fish than either Site or reference fish. Additionally, a statistically high occurrence of abnormal swimming behavior was observed in fish from Site locations compared to reference locations (Golder 2014). The most common swimming-related abnormalities observed were related to physical deformities that inhibited normal swimming behavior.

Gross Pathology. Preserved study specimens were sent to Northwest ZooPath for external examination for abnormalities. A detailed summary of gross pathology report findings is presented in Golder (2014). In general, the frequencies of observed abnormalities were low for all study locations. Mold contamination was commonly observed in study eggs, most frequently from LRC. Abnormal egg shapes were common in all groups likely due to a combination of contact with substrate, bacterial or mycotic infection, and/or decomposition. Most alevin abnormalities were attributed to trauma and conspecific aggression.

9.4.3 Juvenile Trout Toxicity Test

In this study, juvenile trout were placed in cages in both onsite and reference streams to determine if there was a significant difference in growth or survival.

9.4.3.1 Study Design

Exposure Method

Juvenile native cutthroat trout were obtained from the Montana Fish Wildlife and Parks Murray Springs fish hatchery in Eureka, Montana. Trout were placed in cages (15 organisms per cage). Cages used in the study were constructed wooden boxes (13 inches [in] tall x 10 in wide x 12 in long) with metal mesh on the bottom and sides and a solid top. Floats were attached to the sides of the box to keep it suspended in the water column (**Figure 9-13**).

Exposure Locations

A total of six cages were placed in LRC, two cages each at stations LRC-2, LRC-5, and LRC-6 (see **Figure 2-1**). Likewise, a total of six cages were placed into the gravel of reference streams, three cages each at station URC-2 and NSY (see **Figure 2-1** and **Figure 2-9**, respectively). The creek locations for cage deployment were selected to occur in natural pools. In some cases, boulders, rocks, or logs were placed upstream to decrease flow through the cage (**Figure 9-13**).

Field Observations

Juvenile trout were deployed into the streams on May 11, 2012. The cages were left in place for approximately 30 days (the study was terminated on June 13, 2012 for the LRC stations and

June 14, 2012 for the reference stations). Cages were checked and cleaned every day; fish were fed each day and any dead fish were removed. In addition, stream flow, DO, and water temperature were measured and recorded.

Laboratory Swimming Observations

At the end of exposure, the cages were removed from the streams and transported in site water to an onsite laboratory where all living trout were transferred into aquaria. After a brief acclimation period, the swimming behavior of the trout was observed for 30 minutes. Then, the fish were sacrificed and the weight and length of each fish was recorded. Each fish was then placed in preservative for transport to a histological laboratory for external examination.

9.4.3.2 Exposure Conditions

Data for flow (see **Figure 9-5**) and temperature (see **Figure 9-6**) that were measured during the 2012 trout toxicity tests were presented previously in Section 9.4.1.2.

Caged fish are exposed to LA primarily through surface water. For the LRC stations, one surface water sample was collected twice per week at one cage (selected at random) per station. For the reference stations (URC-2 and NSY), one surface water sample was collected once per week at one cage (selected at random) per station. All water samples from site and reference locations were analyzed by EMSL-Libby or EMSL-Cinnaminson for total LA by TEM, treating the water with ozone/ultraviolet prior to analysis to remove any biological material that might cause fiber clumping. **Table 9-17** and **Figure 9-14** present the surface water concentrations[14] of total LA and LA structures longer than 10 μm measured during the juvenile trout toxicity study. As shown, concentrations of LA were higher at the three LRC stations than in the reference stations, with maximum total LA concentrations greater than 30 MFL. Concentrations tended to be highest in early May and decreased over time as flow decreased.

9.4.3.3 Results

A detailed summary of the juvenile trout toxicity test results, as well as a statistical comparison of rates between field and reference locations is presented in Golder (2013b). The interpretation of these toxicity test results is reserved for the ecological risk assessment.

Survival. All data on the occurrence of dead and living organisms recorded during the study were jointly reviewed by Golder and EPA. **Table 9-18** summarizes the data that were agreed upon for the field exposure cages. As shown, survival rates were 100% in LRC fish and 93% in reference fish; these rates were not statistically different (Golder 2013b).

[14] Note: It was determined that some of the water concentrations originally reported by the laboratory were in error. Concentration data presented in Table 9-4 and Figure 9-6 reflect corrected results. See Section 12.6 for additional information.

Size and Weight. **Table 9-19** presents a summary of the mean length and weight of all surviving fish for each station. Surviving trout ranged from 97 to 181 mm in length and 7 to 50 grams in weight in LRC and from 63 to 190 mm in length and 6 to 58 grams in weight in the reference streams. LRC fish were statistically larger than reference fish for both length and weight, perhaps as a result of warmer water temperature and a faster rate of development (Golder 2013b).

Swimming Behavior. A detailed summary of swimming behavior observations for fish from each station is presented in Golder (2013b). In brief, 88% of the fish from LRC stations showed consistently normal swimming behaviors; the remainder exhibited occasional abnormal behavior. For the reference streams, 97% showed consistently normal swimming behaviors; the remainder exhibited occasional abnormal behavior. There did not appear to be increase abnormal swimming behaviors in the fish from LRC (Golder 2013b).

External Examination. Preserved juvenile trout were sent to Northwest ZooPath for external examination for abnormalities. A detailed summary of gross pathology report findings is presented in Golder (2013b). In brief, fish from all locations (LRC and reference) exhibited a range of lesions, including fin lesions (believed to be associated with confined cage conditions and/or conspecific aggression), skin plaques, gill lesions, and asymmetrical atrophy of the fins and operculum. **Table 9-20** summarizes the gross pathological measures that were statistically evaluated. As shown, the presence and severity of fin notching/fraying was statistically higher in LRC fish compared to reference fish (Golder 2013b).

10 Aquatic Community and Habitat Surveys

Another line of evidence that is often relied upon in the evaluation of ecological risks is direct observations of ecological community and habitat metrics. These observations seek to determine whether any receptor population has unusual numbers of individuals (either lower or higher than expected), or whether the diversity (number of different species) of a particular category of receptors (e.g., plants, fish, small mammals, birds) is different at the site than expected (relative to a selected reference area).

At OU3, direct observations (surveys) of the fish and aquatic invertebrate community and stream habitat were made during the 2008 and 2009 field seasons as part of the Phase III sampling program. In addition, a stream pool classification evaluation was performed in 2011 as part of the Phase IV Part B sampling program. An amphibian field study was conducted in 2012 as part of the Phase V sampling program. The following sections summarize the study design and results of the aquatic community and habitat surveys.

10.1 Fish Community

10.1.1 Survey Design

Surveys of fish density and diversity were performed in October of 2008 and September 2009. A total of nine stream locations were evaluated, including two in upper Rainy Creek (URC-1A and URC-2), four in lower Rainy Creek (LRC-1, LRC-2, LRC-3, and LRC-5), one location downstream of the tailings impoundment (TP-TOE2) and at two off-site reference locations (BTT-R1 and NSY-R1) (see **Figures 10-1** and **10-2**).

In 2008, fish were collected using electroshocking equipment. Multiple passes of electroshocking were performed at each sampling location. In 2009, minnow traps were used in addition to the electroshocking passes in an effort to increase the effectiveness of capturing smaller fish. Length, weight, and species type were recorded for each fish collected. Detailed information on the fish community sampling efforts is provided in Parametrix (2009a, 2010).

10.1.2 Results

Table 10-1 summarizes the results from these sampling efforts. In this table, sampling information is provided separately for large fish (length > 65 mm) and small fish (length ≤ 65 mm). After a review of the data for fish caught in the minnow traps, it was determined that the openings on these minnow traps may have been too large (~25 mm in diameter) to effectively capture smaller fish (Parametrix 2010). Therefore, fish from the minnow traps were not included in fish community metrics. **Figure 10-3** summarizes the number of fish caught per acre by species at each sampling station during the first and second electroshocking passes[15]. In this

[15] Because a 3rd electroshocking pass was not performed at all stations, this figure presents the total number of fish per acre based on 1st and 2nd pass electroshocking data only.

figure, larger fish (length > 65 mm) are summarized in Panel A and smaller fish (length ≤ 65 mm) are summarized in Panel B.

Based on the species identification of the larger fish, lower Rainy Creek stations are populated mainly by rainbow trout, though cutthroat trout were present at station LRC-5 in 2009. Cutthroat trout and cutbow trout (cutthroat/rainbow hybrids) tend to be predominant in upper Rainy Creek and Noisy Creek. Bobtail Creek tended to be populated with a mixture of brook trout and rainbow trout. As shown in Panel B of **Figure 10-3,** lower Rainy Creek stations had no fish ≤ 65 mm in length.

Detailed results for the fish community survey are provided in **Appendix D**.

10.2 Benthic Macroinvertebrate Community

10.2.1 Survey Design

Surveys of benthic macroinvertebrate (BMI) density and diversity were performed in 2008 and 2009 at the same site and reference sampling stations where fish surveys were performed (see **Figures 10-1** and **10-2**). At each location, BMI samples were collected using two different protocols. One sample was collected according to EPA's Rapid Bioassessment Protocol (RBP) method (Plafkin et al. 1989; Barbour et al. 1999), and one sample was collected using USFS Surber methods (Barbour et al. 1999). For each sample, invertebrates were identified to the genus level and the relative abundance of each taxon was determined. Detailed information on the BMI sampling efforts are provided in (Parametrix 2009a; 2010).

10.2.2 Results

RBP Samples

The BMI community data collected in accordance with the RBP method are interpreted by combining a number of alternative metrics of benthic community status to yield a biological condition score (BCS), as illustrated in **Figure 10-4**. The BCS values from site stations are compared to BCS values for appropriate reference stations and a biological condition category is assigned for each sampling location.

Table 10-2 and **10-3** present the calculated benthic community metrics, the BCS, and assigned biological condition category for each sampling location for 2008 and 2009, respectively. As seen, in 2008, all lower Rainy Creek stations were ranked as slightly impaired and all upper Rainy Creek stations were ranked as unimpaired relative to the off-site reference areas. In 2009, with the exception of LRC-1 and LRC-2, all upper and lower Rainy Creek stations were ranked as slightly impaired relative to the off-site reference areas. LRC-1 and LRC-2 were ranked as unimpaired.

Surber Samples

As illustrated in **Table 10-4**, the Surber samples are interpreted by calculating a BMI total score from a number of benthic community metrics using a set of scoring criteria established by Montana Department of Environmental Quality (MDEQ) for montane streams (MDEQ 2005). Metrics differ in their possible values ranges as well as in the direction the values move as biological conditions change. To facilitate scoring metric values were transformed into a single scale and assigned a point score between zero to three. A score of three indicates a metric value similar to one characteristic of a non-impaired condition. A score of zero indicates strong deviation from non- impaired conditions and suggests severe degradation of biotic health. **Tables 10-5** and **10-6** present the benthic community metrics and the BMI total score for each OU3 sampling location for 2008 and 2009, respectively. Lower Rainy Creek sampling locations generally had scores at or slightly below the low end of the biological condition scoring range indicting impaired conditions. However, scores for Bobtail Creek (BTT-R1) and upper Rainy Creek (URC-1A) also indicated impaired conditions for some metrics in one or both years.

10.3 Habitat Assessment

10.3.1 Survey Design

Because variations in habitat can contribute to differences in aquatic populations between stations, a habitat assessment was completed at each aquatic community survey location using procedures from EPA's RBP method (Plafkin et al. 1989; Barbour et al. 1999). Ten alternative measures of habitat quality were combined to yield an overall habitat quality score (HQS) for each sampling location that reflects overall habitat quality. For each site sampling location, a relative score (percent of reference) was also calculated. This relative score indicates how closely habitat quality was matched to the reference station.

10.3.2 Results

Tables 10-7 and **10-8** present the HQS for each metric, the overall HQS, and assigned habitat ranking for each sampling location for 2008 and 2009, respectively. As seen, habitat quality at site stations was ranked as suboptimal to optimal, with HQS values tending to be fairly similar across the sampling locations (HQS values for lower Rainy Creek ranged from 120 to 169). Station LRC-1 had the lowest HQS in both 2008 and 2009. LRC-1 is located just below the Mill Pond in Rainy Creek, and scored lower than other stations for available cover, depth, and channel integrity. HQS values for reference stations ranged from 161 to 165 and were similar to upper Rainy Creek stations.

10.4 Stream Pool Assessment

In 2011, the Phase IV Part B data collection efforts included efforts to better characterize the habitat suitability of site streams for fish.

10.4.1 Sampling Design

In addition to surface water LA concentration data (see Section 2.4), the Phase IV Part B study included the collection of stream pool characteristics in OU3 to provide information on habitat factors that may influence fish populations. In small streams, the high temperature in water during the summer is an important factor in determining habitat suitability for fish. Access to deeper pools, where water is cooler, is critical for fish to escape excess heat in the summer, and also to prevent freezing in the winter. Although stream habitat and surface water temperature data were collected in earlier investigations, additional surface water temperature data and more detailed characterization data of the in-stream pools were needed to utilize habitat suitability index (HSI) models for cutthroat and rainbow trout to evaluate the suitability of Rainy Creek to support and sustain fish populations (Hickman and Raleigh 1982; Raleigh *et al.* 1984) and to assess whether habitat factors are influencing fish populations in Rainy Creek. HSI models for salmonids use estimates or measurements of 16 different habitat variables to evaluate habitat suitability over all life stages. The Phase IV Part B habitat data were collected to provide information for HSI model variables V_1 (average maximum water temperature) and V_{15} (pool class rating).

To ensure that the reaches evaluated in the stream pool assessment were comparable to the fish community metrics collected in 2008 and 2009, the same nine reaches sampled for the fish community evaluations were evaluated in the stream pool assessment (see **Figure 10-1**). The stream pool assessment was conducted at seven stream locations in OU3, including two in upper Rainy Creek (URC-1A and URC-2), four in lower Rainy Creek (LRC-1, LRC-2, LRC-3, and LRC-5), and one downstream of the tailings impoundment (TP-TOE2). Two reference locations in the vicinity of OU3 were also evaluated, including one location on a tributary to Bobtail Creek (BBT-R1) and another location on Noisy Creek (NSY-R1) (see **Figure 10-2**). Global positioning system (GPS) coordinates for each stream reach are provided in **Table 10-9**.

In order to ensure that the maximum pool temperature was captured, pool temperatures were continuously monitored at one-hour intervals using a temperature data logger during the warmest portion of the year (i.e., summer months). Temperature monitoring began in June 23, 2011 and extended through October 4, 2011. Temperature monitoring data were collected from the deepest pool within each reach.

The stream pool assessment was performed in September 2011, when stream flows were at their lowest. For each reach, each identified pool was assigned a pool class based on its depth and size (length, width) as follows:

Pool Class	Description
1	Large[16] and deep. Pool depth and size are sufficient to provide a low velocity resting area for several adult fish. More than 30 percent of the pool bottom is obscured due to depth, surface turbulence, or the presence of structures, for example, logs, debris, boulders, or overhanging banks and vegetation. The pool depth is $\geq$ 1.0 meters deep (in streams < 5 meters wide). Note: Rainy Creek averages < 2 meters in width.
2	Moderate size and depth. Pool depth and size are sufficient to provide a low velocity resting area for a few adult fish. From 5 to 30 percent of the pool bottom is obscured due to depth, surface turbulence, or structures. Typical second class pools are large eddies behind boulders and low velocity moderately deep areas beneath overhanging banks and vegetation. Pool depth may range from 0.3 meters to <1.0 meters.
3	Small or shallow or both. Pool depth and size are sufficient to provide a low velocity resting area for one or two adult fish. Cover, if present, is in the form of shade, surface turbulence, or very limited structure. Typical third class pools are wide, shallow pool areas of streams or small eddies behind boulders. Virtually the entire bottom area is discernable. Pool depth is <0.3 meters.

Then, each reach was assigned a pool class rating (A, B, or C) depending upon the surface area coverage of each pool class as follows:

A: > 30% of the reach is comprised of Class 1 pools
B: > 10% to < 30% Class 1 pools, or > 50% Class 2 pools
C: < 10% Class 1 pools and < 50% Class 2 pools

The stream pool assessment and pool temperature monitoring effort was conducted by Anchor QEA, LLC (a subcontractor to Remedium). Data from this study were reported in the *OU3 and Reference Stream Pool Assessment Data Report* (Anchor QEA, LLC [Anchor QEA] 2011). Major findings are summarized below.

10.4.2 Pool Temperature Monitoring Results

Figure 10-5 presents the stream pool temperature monitoring results for each reach and **Table 10-10** presents summary statistics of these results. **Table 10-11** presents stream pool temperature monitoring results by month. Based on a review of the pool temperature data collected for this study, the following observations are noted:

- There are clear differences in stream pool temperatures when comparing the different stream locations. The upper Rainy Creek locations (shown in Panel A of **Figure 10-5**) are cooler than the lower Rainy Creek locations (shown in Panel B of **Figure 10-5**). The

[16] Although the pool class descriptions use size descriptors of "large", "moderate", and "small", the HSI models do not specify any areal requirements for pool size.

reference site in Bobtail Creek (BTT-R1) is much warmer than the reference site in Noisy Creek (NSY-R1).

- Maximum temperatures observed in lower Rainy Creek were generally in the 14 to 18°C range, which are within tolerable ranges for cutthroat and rainbow trout (Hickman and Raleigh 1982). Maximum temperatures observed at Bobtail Creek (~20°C) would be less suitable for cutthroat trout.

- The locations not influenced by an upstream pond (NSY-R1, URC-1A, URC-2, and TP-TOE2) tend to have cooler temperatures than the stream locations affected by an upstream pond.

- The warmer pool temperatures in lower Rainy Creek and Bobtail Creek are likely due to the ponds located above these sites. The cooler pool temperatures measured at the upper Rainy Creek sites are likely due to groundwater sources recharging the stream water (Anchor QEA 2011).

- Riparian cover did not appear to be an important factor in measured pool temperatures (Anchor QEA 2011).

10.4.3 Stream Pool Assessment Results

An expanded stream survey area (i.e., stream reach length was extended 10 meters in each direction) was used in conducting the pool size assessment; however, for NSY-R1 and URC-1A, the stream reach evaluated for the pool size assessment was expanded even further upstream to include the deepest pool used in the temperature assessment. In this evaluation, pool lengths, widths, and depths[17] were measured and the length and average widths of each stream reach were calculated. **Figure 10-6** presents the pool area coverage (in percent) stratified by pool class for each stream reach. **Table 10-12** summarizes stream pool area measurements and classifications. Based on a review of the pool size characterization data for this study, the following conclusions can be drawn:

- Only one stream location (reference area NSY-R1) had a class 1 pool. There was only a single class 1 pool noted for this reach.

- With the exception of BTT-R1, all locations were dominated by class 2 pools.

- Reference site BTT-R1 had the least amount of area covered by pools. The upper Rainy Creek site, URC-1A, had the most area covered by pools.

- Note: At the time of the pool size assessment at BTT-R1, the field teams noted that there were some signs of scouring that were not present when the pool temperature logger was placed. The scouring implies that there was an increase of flow. It is believed that

[17] Pool depth was calculated by subtracting the depth of the pool tail crest from the maximum pool depth.

there was a release of water from the private pond upstream of BTT-R1 (Anchor QEA 2011).

10.5 Phase V, Part B Resident Trout Study

The objective of this study was to determine whether the frequency and severity of lesions or abnormalities in fish in lower Rainy Creek is higher than fish from reference creeks. A detailed summary of the DQOs for resident trout study are presented in Section 6 of the *Phase V, Part B: 2012 Ecological Investigations SAP/QAPP* (EPA 2012b), and the detailed field protocol is presented in Appendix A.4 of the SAP/QAPP. Key study design features and the results of the resident trout study are summarized below.

10.5.1 Sampling Design

Data are not available to indicate whether there are any potential age-dependent effects of LA on fish. Because young fish are often more sensitive to some metals than older fish and the density of young of the year (YOY) in LRC appears to be much lower than in reference streams (based on earlier population studies), it was decided that this study should focus on young fish. Target species included resident fish of LRC: rainbow, cutthroat, and cutbow trout. The target size range was fish less than 100 mm in length, which would include YOY as well as some juvenile fish from the previous year's hatch. The goal was to collect fish that were representative of two size classes (≤ 65 mm and 65 to 100 mm).

Resident trout were captured in LRC and in two reference areas, URC and NSY. LRC was considered the optimum location for collection of resident fish that have been exposed to LA. A creek reconnaissance was conducted immediately before the study to identify any station reaches that required adjustment and to identify logistics and deployment activities needs. The capture time was moved from late September to early August in LRC in order to increase the likelihood of capturing fish in the smaller size class. This change was made because warmer water temperatures in LRC increased fish growth and development.

Resident trout were captured using electro-shocking at LRC, URC, and NSY in early August 2012 (between August 1 and 6, 2012). Minnow traps were also deployed, but were largely unsuccessful in capturing any fish. Within LRC, fish were collected at multiple stations, including locations where previous population studies have been performed. Sampling was conducted at stations LRC-2, LRC-3, LRC-4, LRC-5, and TP-TOE2 (see **Figure 10-1**) and at reference stations URC-2, URC-1A, and NSY-R1 (see **Figures 10-1** and **Figure 10-2**). To the extent feasible, approximately equal numbers of fish were collected at each station to help ensure the data set was representative and was not unduly influenced by any individual station.

Captured fish were examined in the field for external parasites or other external gross abnormalities. Lengths of collected fish were measured in the field, from the tip of the snout to the tip of the top lobe of the caudal fin (to the nearest mm) and recorded in the field notebook

and forms. Only cutthroat, rainbow, and cutbow trout less than 100 mm in length were kept; all other fish were released. Collected fish were kept in plastic containers filled with cold water from their respective creek until transported to the onsite laboratory for processing and preservation in accordance with the processing methods identified in the governing SAP/QAPP (EPA 2012b). Processing in the offsite laboratory included: fish euthanization, recording the weight of each fish, verification of field collected length measurement, and preserving the organism for examination by the histopathologist.

Sixty captured fish were euthanized, preserved in 10% neutral buffered formalin, and shipped to the Northwest ZooPath pathology laboratory in Monroe, Washington for external and histological examinations. External examinations focused on abnormalities present on the head, fins, skin, and gills. Particular attention was paid to any abnormalities of the gills and lateral line. If tumors or other anomalies were identified, these tumors/abnormalities were excised, sectioned, stained, and examined microscopically by the pathologist.

No surface water samples were collected as part of this study. This is because a single water sample collected at the time of fish collection would not necessarily reflect the concentrations to which the fish have been exposed to for several months or longer.

10.5.2 Results

Detailed study results are presented in the *Data Report: 2012 Resident Trout Study* (Golder 2014a) and summarized below.

The number and types of fish caught at each location for both size classes is summarized in **Table 10-13**. As seen, more fish were caught in the reference locations (URC, NSY) than in LRC. No fish were caught at station LRC-4 or LRC-5. Cutthroat, rainbow, and cutbow trout retained for evaluation ranged from 42 to 100 mm in length and weighed between 0.6 and 9 grams (see **Table 10-13**).

All fish were examined initially by Northwest Zoopath for gross external lesions or abnormalities under a dissecting light microscope, with special attention to gill tissue and lateral line. The pathologist identified gross and histologic lesions in fish collected from all locations, including both LRC and the reference locations); most of these lesions occurred on the fins, skin, and gills (see **Table 10-14**). According to the pathologist, all the lesions observed are commonly encountered in other fish populations and are attributable to a combination of trauma, stress, or suboptimal water quality (Golder 2014a).

Based on the results of the external examination, additional histological examinations were performed on 18 fish, representing a range of observed gross external lesions, including fish with identified abnormalities and fish with no lesions (used as histology control references). These fish were sectioned transversely at four locations to include the head and rostral aspect of the coelom and body, such that the gills, cranial line, lateral line, fins, and skin could be

examined symmetrically for microscopic lesions, and to evaluate the pathogenesis of any observed macroscopic lesions. Observed abnormalities were scored based on severity (e.g., inflammation, hemorrhage, edema, necrosis, etc.) and coverage. Scoring was focused on the nose, dorsal, lateral, and opercula regions of the head skin, cranial line, cornea, brain, gills, oral and nasal mucosa, lateral, dorsal, and ventral areas of the trunk skin, lateral line, fins, and skeletal muscle. The lesion severity scores were generally higher in fish from the NSY reference location (see Table 3-7 of Golder 2014a).

The pathologist found that histologic lesions were more extensive in the gills and skin than were apparent from gross (external) examination, suggesting that gross lesion assessment is not a sensitive means of identifying lesions in these fish. However, no primary infectious agents or deposition materials were identified histologically that would account for the lesions. In addition, no unique lesion morphology was identified to suggest that asbestos was a contributing factor to lesion development fish from LRC (Golder 2014a).

A comparison of the frequency of occurrence and severity of fish abnormalities between LRC and reference locations determined that neither the occurrence nor severity of external abnormalities was found to be statistically higher in LRC fish compared to reference location fish (see **Table 10-15**). There was a statistically higher occurrence of histological abnormalities of the skeletal muscle in LRC fish compared reference (see Table 3-6 in Golder 2014a). The pathologist attributed these abnormalities in the skeletal muscle to the capture method (electro-shock) used; however, since all fish were captured by electro-shocking the basis of this assertion is unclear.

10.6 Phase V, Part B Amphibian Field Study

The goal of this study was to determine if the frequency of lesions or abnormalities is higher in amphibians developing in onsite locations containing LA than in amphibians developing in (uncontaminated) reference locations. A detailed summary of the DQOs for the amphibian field study are presented in Section 4 of the *Phase V, Part B: 2012 Ecological Investigations SAP/QAPP* (EPA 2012b), and the detailed field protocol is presented in Appendix A.2 of the SAP/QAPP. Key study design features and the results of the amphibian field study are summarized below.

10.6.1 Sampling Design

Study Areas

A field reconnaissance was performed in March 2012 to determine candidate locations for collecting developing amphibians. Onsite areas selected included: Carney Creek Pond, Fleetwood Creek Pond, Mill Pond, and the Tailings Impoundment (Golder 2012) (see **Figure 10-7**). Reference areas selected included: Bobtail Pond, Banana Lake, and Tepee Pond 1 (Golder 2012) (see **Figure 10-8**). Prior to use as a reference location, sediment samples were collected

from each candidate reference pond for analysis of asbestos and non-asbestos analytes to ensure that the ponds were not contaminated. Candidate reference areas included: Bobtail Pond 1, Bobtail Pond 2, Banana Lake, Shrieber Lake, Tepee Pond 1, and Tepee Pond 2. Reference areas were selected based on the analytical results and general habitat quality.

Environmental Characterization

The amphibian field study was conducted by Remedium's contractor (Golder) in accordance with the EPA-developed SAP/QAPP (EPA 2012b). During the study, the field teams visited the onsite and reference locations twice a week to check if specimens from each developmental window were available (see below). The exact time that amphibians breed and their eggs begin development depends on many environmental factors, especially temperature. During each of the bi-weekly visits, water temperature was measured and recorded.

Surface water samples were collected weekly once egg masses were confirmed to be present starting on May 24, 2012 and ending on August 31, 2012. Surface water sampling was performed in basic accordance with the OU3-specific SOP No. 3, *Surface Water Sampling*, using the direct sampling methods. Water samples were hand-delivered to EMSL-Libby for analysis of LA by TEM, treating the water with ozone/ultraviolet prior to analysis to remove any biological material that might cause fiber clumping.

Because sediments are not expected to vary substantially over time, two samples of sediment were collected for analysis of LA, the first sample near the beginning of the study (May 5, 2012) and the second sample near the end of the study (October 9, 2012). Sediment sampling was performed in basic accordance with the OU3-specific SOP No. 5, *Sediment Sampling*. In brief, at each pond surficial sediment was collected from the pond edge at multiple points around the pond and composited into a single sample. After being processed (dried, sieved, ground) by the Troy SPF, sediment samples were sent to EMSL-Libby for analysis of LA by PLM-VE. There were not any coarse fractions for sediment samples. Non-asbestos analyses of sediment were performed by ELI.

Detailed analytical results (asbestos and non-asbestos) for all Phase V Part B samples are provided in the OU3 project database (see **Appendix A**).

Amphibian Developmental Stages

Because no information has been located on the potential effects of asbestos on amphibians, it is not known what life stage is likely to be most sensitive. Consequently, the field study evaluated the full developmental period from egg mass through metamorphosis. The frequency of specimen collection from each developmental window (see below), was dependant upon meteorological conditions and specimen availability. Developmental stages were stratified into four windows, as follows:

- Egg mass
- Embryo-larval (Gosner stages 21-25, see **Figure 10-9**)
- Hind limb development completion (Gosner stages 37-40, see **Figure 10-9**)
- Metamorphic completion (post-climax) (Gosner stage 46, see **Figure 10-9**)

Amphibian Measurement Endpoints

Measurement endpoints for each developmental window included external physical examination for abnormalities in all specimens and all life stages, regardless of species. In addition, necropsy was performed in all newly metamorphosed specimens. Histopathology was performed for the one species with the most complete data set at each site[18], with special attention to gills, mouth, skin, and gonad tissue. Others tissues were examined, depending on the outcome of external examination and necropsy, focusing on specimens with developmental anomalies.

10.6.2 Results

Detailed results of the amphibian field study are presented in *Data Report: 2012 Field Collection, Examination and Pathology of Amphibian Species* (Golder 2014b) and are summarized briefly below.

10.6.2.1 Asbestos Results

Surface Water

As seen in **Table 10-16** and in **Figure 10-10**, LA was detected in the majority of samples collected in onsite surface water samples. Total LA concentrations ranged widely from less than 1 MFL to 109 MFL (observed at Fleetwood Creek Pond). Concentrations of LA for structures longer than 10 µm in length ranged up to 28 MFL (also observed at Fleetwood Creek Pond). Total LA was detected at a concentration of less than 1 MFL in one out of the six reference surface water samples collected; this sample was collected from Banana Lake.

Sediment

Table 10-17 (Panel A) presents the results for PLM-VE results for sediment samples collected prior to the amphibian field study. As shown, LA concentrations ranged between <1% and 10% LA in the onsite ponds; no asbestos was detected in any of the offsite ponds. **Table 10-17** (Panel B) presents the results for PLM-VE results for sediment samples collected during the amphibian

[18] Of the multiple species that were collected, the one species that had the most complete number of Gosner stages represented were selected for histopathology examination.

field study. As shown, LA concentrations ranged between trace and 5% LA in the onsite ponds; no asbestos was detected in any of the offsite ponds.

10.6.2.2 Non-Asbestos Results

Surface Water

Non-asbestos analyses (e.g., metals) were not performed for surface water samples collected for this study. Temperature measurements were taken at each pond visit (at least two per week) using a handheld infrared digital thermometer. As seen, there was over a 10 degree C difference between the minimum and maximum measured temperatures. Average temperatures were the lowest at Banana Lake and highest at Tepee Pond, both reference areas. Generally site and reference area surface water temperatures were similar. A summary of the measured temperatures are presented in **Table 10-18**.

Sediment

Table 10-19 (Panel A and Panel B) present summary statistics for detected non-asbestos chemicals in sediment in onsite and reference study areas, respectively. As seen, various metals were detected in control and reference sediments. PAHs, PCBs, and pesticides were not detected above reporting limits. Chemicals detected in site sediments included metals and low levels of petroleum hydrocarbons.

10.6.2.3 Summary of Amphibian Data

Table 10-20 summarizes the number, species, and developmental stage of amphibians collected at each location. As seen, all species and developmental stages were not able to be collected at every pond. For example, tree frog eggs were only collected from Carney Creek Pond and no spotted frog eggs or western toad eggs were collected from any area. A total of 315 amphibian specimens from reference ponds and 477 amphibian specimens from Site ponds were collected. Tree frogs, spotted frogs, and western toads were collected from all areas except Mill Pond, where no amphibians were collected. Western toads were the least frequently collected amphibian and collected only from the Carney Creek Pond, Tailings Impoundments, and the Tepee Pond.

Collected organisms were euthanized and shipped FEL in Stillwater, Oklahoma for external (all specimens) and internal (metamorphosed specimens only) examinations to determine the occurrence of any gross abnormalities. Following this evaluation, metamorphosed specimens were sent to Northwest ZooPath in Monroe, Washington for complete histopathology examinations.

Detailed results of the external and internal examinations are presented in *Data Report: 2012 Field Collection, Examination and Pathology of Amphibian Species* (Golder 2014b) and are summarized briefly below.

External Examinations

External examinations focused on the eyes, mouth, torso, and hind limbs. No malformations were found in any of the larval amphibians. One malformation (i.e., missing or underdeveloped long bones) was observed in a northern tree frog metamorphosed specimen collected from Fleetwood Creek Pond. However, upon examination, this malformation was determined to likely have resulted from predation, rather than congenital malformation.

Statistical evaluations of size (weight, snout-vent length, and hind limb length) performed by Golder indicated some differences between the Site and reference amphibians, but did not present any consistent trends between locations (Golder 2014b). Potential differences in growth endpoints seen in earlier Gosner stages were minimized by Gosner stage 46 (metamorphosis). Amphibians from LA-containing ponds and reference ponds were all normal and healthy appearing with variable growth and developmental patterns consistent with normal wild field amphibian populations. Differences observed in developmental and growth rates may have been related to the effects of differing habitat and climate between locations (Golder 2014b).

Histological Evaluation

A total of 145 metamorphosed tree frogs and spotted frogs underwent histological examination by a veterinary pathologist. Tissue sections from metamorphosed organisms were examined using light microscopy. Observed lesions were documents and scored by distribution and severity. A total of 48 different tissues were evaluated, although not every tissue was examined in every frog. Average lesion scores were highest in spotted frogs from Banana Lake and Tepee Pond (reference ponds). Observed body and tissue lesions were described as primarily inflammatory and parasitic. The only potentially toxicant-induced lesion observed in the study were those seen in the liver. However, these lesions were considered attributable to sources other than toxicant induced for example hypoxia or the euthanasia agent (MS-222). No lesions specifically attributed to asbestos were seen in study frogs (Golder 2014b).

Although no lesions specifically attributable to LA were noted by the pathologist in any of the study frogs, statistical analyses were conducted on the frequency and severity of the observed lesions and compared between Site and reference ponds. A higher occurrence of lesions in some tissues (e.g., dorsum skin, coelomic cavity, liver, and gall bladder) in Site spotted frogs compared to reference frogs was noted; however, all the lesions were attributed to parasitism (Golder 2014b).

Conclusion

Overall the study found that early-stage growth patterns for amphibians were similar for site and reference areas. In addition, higher frequencies of gross abnormalities or histological lesions attributable to LA exposure were not observed in site samples (Golder 2014b).

11 Small Mammal Community Surveys

As noted above, direct observations of the ecological community at a site are often used as one line of evidence in the assessment of potential ecological risks. In the case of small mammals, because there are no accurate and representative data on measures of LA exposure (dose) of small mammals to site media, and because there is no reliable dose-response relationship for LA small mammals, the ecological risk assessment will rely on small mammal community surveys to provide information on potential effects at the OU3 site (EPA 2008d).

Direct observations (surveys) of the small mammal community were made during the 2009 field season as part of the Phase III sampling program. The following sections summarize the study design and results of the small mammal community surveys.

11.1 Survey Design

Revision 1 of the Phase III SAP (EPA 2009b) summarized several alternative strategies for the investigation of potential risks to small mammals that were considered by EPA. After deliberation with the OU3 BTAG, it was determined that the Phase III small mammal community survey would seek to evaluate if individual mammals from an LA-contaminated forested area have a higher incidence and severity of histological lesions and/or gross deformities than mammals from a reference area.

In order to maximize the probability of detecting *in-situ* effects if they are present, the small mammal survey was performed at a location in the forest area where exposures to asbestos were expected to be highest based on the LA levels in forest duff, soil, and tree bark at OU3 (see **Figure 6-20**). Based on the duff data, a small mammal collection polygon for the forested area was established, which was bounded by four sampling locations where some of the highest LA concentrations have been measured in duff:

- SL-15-02 – LA concentration = 3.65% (2,230 Ms/g)
- SL-45-02 – LA concentration = 1.74% (3,082 Ms/g)
- SL-45-03 – LA concentration = 4.27% (2,630 Ms/g)
- SL-75-03 – LA concentration = 3.52% (3,146 Ms/g)

This set of four stations bounds a triangular polygon (see **Figure 11-1**) that covers an area of about 716,000 square meters (m^2) (72 hectares). After a site reconnaissance effort in June 2009 (Golder 2010), trapping locations for the selected site area and a reference area in the Kootenai National Forest near Sheldon Mountain were identified (see **Figure 11-2** and **Figure 11-3**, respectively). **Table 11-1** provides coordinates of the OU3 and reference locations evaluated in this study.

Detailed information on the small mammal survey design is provided in Revision 1 of the Phase III SAP (EPA 2009b). In brief, trapping was planned for late summer during the driest time of the season and when small mammal populations are at peak levels to maximize potential LA releases from soil and Target animals were deer mice and southern red-backed voles. These

animals were targeted because they have small home ranges, forage on the ground, and have small body weights, and were the most common ground-foraging small mammals in Lincoln County. The number of animals desired was 30 animals per species per location (i.e., OU3 and reference), for a total of 120 animals per area. Equal number of males and females were desired to the extent possible.

Trapping and necropsy was performed between August 27 and September 2, 2009 by Golder (subcontractor to Remedium). Sherman live traps and Havahart® live traps were set one to three hours before dusk along trap lines at spacing intervals appropriate to field conditions and at least 15 feet apart along logging or forest roads. The steepness of the terrain and shrub density affected trap placement in some areas. Traps were checked one to two hours after sunrise and live target animals were transported to the field laboratory for field processing. Non-target species were released. After recording trap and animal identification information, the animal was euthanized. Each animal was examined for abnormalities and sex, and was measured, weighed, and photographed. Animal were stored on wet ice in a cooler until necropsy was performed. Eyeballs were removed for later use in aging. Animals were opened and the body cavity and viscera were photographed. Internal organs were examined for abnormalities and lesions. Tissue samples for possible future LA analysis were harvested and preserved by placement into formalin fixative for histopathological examination. Target tissues for collection for histopathological examination included: complete pulmonary tract, complete gastrointestinal tract, thyroid, and adrenals.

Details of the field collection efforts for the small mammal survey, including all field documentation, are summarized in the *Summer 2009 Small Mammal Data Collection Program* final data report (Golder 2010). A summary of study findings are presented below.

11.2 Results

A total of 72 deer mice were collected as part of the small mammal survey, 34 mice from the reference sites and 38 mice from the OU3 sites. No voles were collected from either location. The overall female-to-male ratio for the animals captured from the reference area was 1.8, whereas this ratio was 0.8 for OU3. However, sex ratios between transects were variable at both the reference area and at OU3. Based on the average dry eye lens weight, the average mouse age ranged from 96 to 316 days (i.e., three to over ten months in age). A summary of the species and number of animals captured at each location is presented in **Table 11-2**.

Histological examination found no evidence of asbestos pathology in any target tissues or submitted lesions. Observed lesions were attributed to parasite- and disease-related inflammation by the pathologist. The pathologist also indicated that all mice had recognizable and abundant fat stores, which was indicative of adequate nutritional status. None of the mice had evidence of prominent stress response in the lymphoid tissues or the adrenals examined.

12 Quality Assurance/Quality Control

The purpose of this section is to describe the quality assurance (QA) procedures that have been established to govern the collection and analysis of environmental samples at OU3 to ensure resulting data are of high quality. This section also summarizes the results for a variety of different types of quality control (QC) samples that have been collected across the various sampling programs that provide information on the accuracy, precision, and reliability of reported results.

12.1 Field Quality Assurance Activities

12.1.1 General

Field QA activities include all processes and procedures that have been designed to ensure that field samples are collected and documented properly, and that any issues/deficiencies associated with field data collection or sample processing are quickly identified and rectified. Detailed information on field QA activities can be found in the investigation-specific SAP/QAPPs. These SAP/QAPPs are developed by EPA technical support contractors and implemented by Remedium field contractors. The following bullets summarize the components of the field QA program implemented at OU3.

- **Field Team Roles/Responsibilities –** There are a variety of field personnel involved in the sampling investigations for OU3 and each individual has assigned roles and responsibilities. The field team leader (FTL) oversees all sample collection activities to ensure that governing documents are implemented appropriately. The field QA manager is responsible for ensuring that all field efforts are conducted in accordance with appropriate QA guidelines.
- **Field Team Training** - Individuals involved in the collection, packaging, and shipment of samples must have appropriate training, including Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) and relevant 8-hour refreshers, respiratory protection, and asbestos awareness training.
- **Orientation –** Field personnel are required to attend an orientation session with the field Health and Safety (H&S) manager, as well as an orientation session on sample collection techniques.
- **Investigation-Specific Documentation** - Field personnel are required to review and understand all applicable governing documents associated with the sampling investigation, including the SAP/QAPP, all associated SOPs, and the applicable Health and Safety Plan (HASP).
- **Readiness Reviews** - Meetings are conducted prior to beginning field sampling activities to discuss and clarify the objectives, equipment and training needs, field SOPs, QC samples, and H&S requirements for each investigation.

- **Field Documentation Review –** Field documentation is completed by field staff using investigation-specific field forms. These field forms provide a standardized method of documenting sample information generated in the field. Field documentation is reviewed on a regular basis to ensure the accuracy of the recorded sample information.
- **Equipment Maintenance/Calibration –** All field equipment is maintained in accordance with manufacturer specifications and OU3-specific SOPs. For air samples, each air sampling pump is calibrated to the desired flow rate using a primary calibration standard prior to sample collection.
- **Equipment Decontamination –** Field equipment used in sample collection is decontaminated in accordance with OU3-specific SOPs. Any disposable equipment or other investigation-derived waste (IDW) is handled in conformance with SOP requirements.
- **Sample Custody/Tracking** - All samples collected at OU3 are tracked and managed in accordance with OU3-specific SOPs for sample custody and tracking using appropriate COC forms.
- **Field QC Samples** - A variety of different types of field QC samples have been collected as part of the investigations conducted at OU3. These QC samples provide information on potential contamination arising from sample collection methods as well as information on result precision. (See Section 12.4.1 for a detailed discussion of field QC results.)
- **Modification Documentation –** Major deviations to the SAP/QAPP that modify the sampling approach and associated guidance documents are recorded on a field record of modification (ROM) form. These ROMs are reviewed and approved by the EPA Regional Program Manager (RPM).

12.1.2 Field Oversight

Because field sampling activities at OU3 are performed by Remedium contractors, an important component of the field QA program is field oversight. From 2007 to 2009, field oversight was provided by EPA's contractor, CDM Smith. Starting in 2010, field oversight has been performed by EPA's contractor, HDR Engineering, Inc. (HDR).

Prior to initiating oversight activities, CDM Smith staff associated with the oversight activities reviewed all governing investigation-specific documents and prepared blank audit checklists to be completed during the field oversight activities.

In 2007, CDM Smith performed a field audit of ambient air station installation and sample collection for water, sediment, mine waste, forest soil, duff, tree bark samples collected as part of Phase I. A total of 10 audits were conducted from October 3 to October 18, 2007. In 2008, CDM Smith performed a field audit of flume construction, flow measurements, and collection of surface water and sediment samples for the Phase II, Part A investigation. A total of 41 field audits over 14 days in April 2008 were completed. Although some minor deviations were noted by the field auditor, there were no significant departures from the SAP/QAPP or SOPs in

regards to sample collection or documentation that were noted in any of the CDM Smith field oversight efforts.

In 2010, two HDR field oversight activities were performed during Phase IV, Part A ABS efforts in July and August. In 2012, HDR field oversight activities were performed in April and May during the Phase V, Part B surface water sampling efforts and in September during the Phase V, Part A during the Kootenai surface water, sediment, and recreational visitor ABS activities. Oversight was conducted according to the Oversight Plans for each activity. HDR prepared the individual Oversight Plans to verify the activities occurred as detailed in the corresponding investigation-specific SAP/QAPPs. Photographs of the sampling activities, supporting figures, and field notebook documentation of HDR's oversight activities are presented at the end of each Oversight Report. In general, oversight activities were consistent with the strategy presented in the Oversight Plan. HDR noted some minor deviations, but the procedures and protocols outlined in the investigation-specific SAP/QAPPs were generally followed, and the overall program intent was met.

12.2 Soil Preparation Laboratory Quality Assurance Activities

12.2.1 General

Prior to 2012[19], all soil, mine waste, and sediment samples collected from OU3 were sent to the CDM Smith CSF in Denver, Colorado for preparation prior to analysis by PLM. The *CSF Soil Preparation Plan* (CDM Smith 2004) served as the guidance document for all activities at the CSF. Beginning in 2012, soil/sediment samples collected from OU3 were sent to the SPF in Troy, Montana for preparation prior to analysis by PLM. The *SPF Soil Sample Preparation Work Plan* presented in Appendix F of the *Troy Asbestos Property Evaluation Work Plan* (Tetra Tech, EM Inc. 2007) serves as the guidance document for all activities at the CSF. The purpose of the soil preparation plans (SPPs) is to provide standard guidance on preparation methods to ensure that these procedures and resulting measurements were scientifically sound and of acceptable and documented quality. The following bullets summarize components of the QA procedures at the preparation laboratories.

- **Personnel Training** - Individuals involved in the processing of samples are required to have read and understood the SPP, all associated SOPs, as well as the facility health and safety plan. In addition, personnel must have appropriate training, including OSHA 40-hour HAZWOPER and relevant 8-hour refresher updates.
- **Documentation Review** – Sample preparation documentation is completed by preparation laboratory staff using Libby-specific forms. These forms provide a standardized method of documenting sample preparation information generated. This documentation is reviewed on a regular basis to ensure the accuracy of the recorded preparation information.

[19] Although the Troy SPF took over preparation for the Libby project in 2009, no OU3 samples required processing until 2012.

- **Equipment Maintenance/Calibration –** All weight scales, ventilation hoods, and drying ovens used in sample preparation are maintained and calibrated in accordance with manufacturer specifications. In addition, the plate grinder is calibrated daily, to verify proper particle size and demonstrate that samples are not being over-processed.
- **Equipment Decontamination –** Sample preparation equipment is decontaminated in accordance with Libby-specific SOP ISSI-LIBBY-01 between each sample.
- **CSF Contamination Monitoring –** The preparation laboratory performs regular contamination monitoring to evaluate worker safety, ensure laboratory cleanliness, and help assess the potential for cross-contamination of samples submitted to the facility.
- **Sample Custody/Tracking** - All samples collected processed at the preparation laboratory are tracked and managed in accordance with COC requirements specified in the SPP.
- **Preparation QC Samples** - A variety of different types of preparation QC samples have been included in the preparation of sample collected as part of the investigations conducted at OU3. These QC samples provide information on potential contamination arising from sample preparation methods as well as information on result precision. (See Section 12.4.2 for a detailed discussion of preparation QC results.)
- **Modification Documentation –** Major deviations from the Libby-specific preparation SOP are recorded on a ROM form. These ROMs are reviewed and approved by the EPA RPM (or their designee).

12.2.2 Audits

EPA's QATS contractor (CB&I, formerly Shaw Environmental, Inc. [Shaw]) performed an audit of the CDM Smith CSF on October 2, 2008. CB&I has performed three audits of the Troy SPF (September 2008, August 2012, and July 2013). Specific activities that were audited included the general laboratory facility, laboratory organization and personnel, general housekeeping, sample receipt and storage, sample preparation procedures, measurements and documentation, sample shipping procedures, and QA/QC procedures.

The 2008 CSF audit report was issued in March of 2009 (Shaw 2009). In brief, a total of 17 observed deficiencies were noted, as compiled from the completed summary on-site audit report, during the 2008 CSF audit (CB&I 2013b). The deficiencies identified during the audits were grouped into eight laboratory process areas. The laboratory process area categories in which the majority of the observed deficiencies occurred included bulk drying, sample receiving, and QA/QC (CB&I 2013b).

For the Troy SPF, a total of eight deficiencies were identified in 2008, ten deficiencies were identified in 2012, and six deficiencies were identified in 2013. The laboratory process area categories in which the majority of the observed deficiencies occurred included bulk drying, grinding and splitting, and QA/QC (CB&I 2013b; 2014). Overall, the total number of deficiencies identified is low and decreased from 2012 to 2013, which suggests that corrective actions taken at the SPF to address previous deficiencies were effective.

12.3 Analytical Laboratory Quality Assurance Activities

12.3.1 General

All laboratories selected for analysis of samples for asbestos are part of the Libby analytical laboratory team. These laboratories have all demonstrated experience and expertise in analysis of LA in environmental media, and all are part of an ongoing Libby-specific QA program designed to ensure accuracy of analytical and consistency of reported analytical results between laboratories. These laboratories are audited by EPA's QATS contractor and the National Institute of Standards and Technology (NIST)/National Voluntary Laboratory Accreditation Program (NVLAP) on a regular basis.

Laboratory QA activities include all processes and procedures that have been designed to ensure that data generated by an analytical laboratory are of high quality and that any problems in sample preparation or analysis that may occur are quickly identified and rectified. The following bullets summarize the laboratory QA procedures that are required of each laboratory that analyzes samples from OU3.

- **Laboratory QA Management Plan** - Each laboratory has developed a laboratory-specific QA Management Plan that provides a detailed description of the procedures and policies that are in place at their laboratory to ensure laboratory quality.
- **Certifications** - All analytical laboratories are subject to national, local, and project-specific certifications and requirements. Each laboratory is accredited by the NIST/NVLAP for the analysis of airborne asbestos by TEM and/or analysis of bulk asbestos by PLM. This includes the analysis of NIST/NVLAP standard reference materials (SRMs), or other verified quantitative standards, and successful participation in two proficiency rounds per year each of bulk asbestos by PLM and airborne asbestos by TEM supplied by NIST/NVLAP.
- **Team Training/Mentoring Program -** Laboratories are required to participate in a training/mentoring program to ensure laboratories can demonstrate the ability to perform reliable analyses at the Site. The training process includes a review of morphological, optical, chemical, and electron diffraction characteristics of LA using site-specific reference materials, as well as training on project-specific analytical methodology, documentation, and administrative procedures used on the Libby site.
- **Technical Discussions/Conferences -** Laboratories participate in regular technical discussions with EPA and their contractors, as well as attend professional/technical conferences. These discussions enable the laboratory and technical team members to have an ongoing exchange of information regarding all analytical and technical aspects of the project.
- **Analyst Training -** All TEM and PLM analysts are required to undergo method-specific training and must understand the application of standard laboratory procedures and methodologies, including the Libby-specific analytical methods. Analysts must

familiarize themselves with -the Libby-specific method deviations, project-specific documents, and visual references.

- **Data Reporting -** Standardized benchsheets and data entry spreadsheets have been developed specifically for the Libby project to ensure consistency between laboratories in the presentation and submittal of analytical data. All analysts are trained in the project-specific reporting requirements and data reporting tools utilized in transmitting results.
- **Laboratory QC Samples** - A variety of different types of laboratory QC analyses have been collected as part of the investigations conducted at OU3. These QC analyses provide information on potential contamination arising from laboratory preparation and analysis methods as well as information on result accuracy and precision. (See Section 12.4.3 for a detailed discussion of analytical laboratory QC results.)
- **Laboratory Contamination Monitoring –** Each analytical laboratory performs regular contamination monitoring to evaluate worker safety and ensure laboratory cleanliness in compliance with their SOPs and certification requirements.
- **Modification Documentation -** Changes or revisions needed to improve or document specifics about analytical methods or laboratory procedures are documented using a ROM form. These ROMs are reviewed and approved by the EPA RPM (or their designee).

12.3.2 Laboratory Audits

Each laboratory conducts internal audits of their specific operations on an annual basis using appropriate checklists in accordance with their laboratory-specific QA Management Plan. As noted above, the laboratories that are part of the Libby analytical laboratory team are also audited by EPA's QATS contractor on a regular basis to specifically evaluate adherence to all Libby-specific analytical requirements. On-site audits are used by EPA to verify samples analyzed by their contract facilities are being processed in accordance with EPA requirements. Each on-site audit involves a review of the general elements of preparation, on-site support, and report generation, which are modified as needed to fit the type of audit being performed.

A series of laboratory audits was performed in April-September of 2008 to evaluate all of the TEM and PLM laboratories that performed analyses in support of OU3. A second round of analytical laboratory audits was performed in June-August of 2012. Another round of analytical laboratory audits was conducted in May-October of 2013.

In addition, a laboratory audit of the Oregon State University (OSU) Aquatic Toxicology Laboratory[20], which performed the site-specific surface water trout toxicity tests for OU3 (see Section 9.1.2), was performed in June 2011. A laboratory audit was also performed in November 2012 of FEL which conducted the amphibian toxicity tests for OU3 (see Section 9.4).

[20] The OSU Aquatic Toxicology Laboratory noted in the QATS audit report and the PERL facility noted in Section 9.1.2 are the same.

Detailed findings for each laboratory audit are documented in separate laboratory-specific audit reports. The overall conclusions of these laboratory audits are presented in CB&I (2013b; 2014) and summarized below.

Analytical Laboratories

A total of 63 observed deficiencies, compiled from the completed summary on-site audit reports, were identified from the on-site audits performed for four different analytical laboratories in 2008 (CB&I 2013b). The deficiencies identified in these laboratory audits were grouped into eight laboratory process areas. The laboratory process categories in which the majority of the observed deficiencies occurred included PLM, sample preparation, sample receiving, and QC/QA; whereas the laboratory process categories with the least frequently occurring deficiencies included TEM, facility, and data management (CB&I 2013b).

EPA requires that laboratories provide responses to on-site audit reports that include the laboratory's proposed corrective action to each of the identified audit deficiencies. Laboratory responses to the 2008 on-site audit reports were received from all the OU3 support laboratories. The laboratory responses provided proposed corrective actions for the identified findings along with objective evidence as applicable. No findings were contested.

A total of 49 deficiencies were identified during the on-site audits of five different analytical laboratories conducted in 2012. These results show that the total number of deficiencies identified in 2012 decreased for all four laboratories audited in both 2008 and 2012, which suggests that corrective action performed in response to previous audit findings were effective (CB&I 2013b).

A total of 19 deficiencies were identified during the on-site audits of four different analytical laboratories conducted in 2013. For the laboratories audited in both 2012 and 2013, all laboratories had fewer deficiencies in 2013 compared to 2013 (CB&I 2014). Again, these findings support the conclusion that corrective actions performed following the 2012 audits were effective.

OSU Aquatic Toxicology Laboratory

The audit of the OSU Aquatic Toxicology Laboratory involved an evaluation of the pilot study protocol for the toxicity test against the procedures used by the laboratory. These included the shipping and receiving of test organisms, standards, and collected samples; the preparation and monitoring (physical and chemical) of test chambers; sample collection; a review of the laboratory's record keeping practices for shipping and receiving, test chamber preparation, and analytical measurements; the availability of written procedures; and the presence of a viable QA/QC program. Several on-site audit deficiencies were identified, including improper COC procedures, inadequate documentation, and deviations from the study protocol and governing SAP/QAPP that were not adequately communicated (CB&I 2013b). As noted in Section 9.1.2 above, there were a number of limitations related to the LA exposures that were also identified with the surface water toxicity test that limit the reliability and usability of the test results.

Fort Environmental Laboratory

The audit of the FEL involved an evaluation of the toxicological study to examine the effects of LA on the complete metamorphosis of amphibians. The on-site evaluation identified only one observation: there was a failure to analyze the laboratory control (inert sand) and reference sediment for the presence of LA prior to initiation of the study (CB&I 2013b). No other deficiencies or deviations were noted.

12.4 Quality Control Results

As discussed above, there are a variety of field QC samples, preparation laboratory QC samples, and analytical laboratory QC analyses are included as part of the sampling investigations performed at OU3. A detailed review and discussion of the results for all QC samples and analyses performed from 2007 to 2012 is provided in the QC summary report for OU3 prepared by EPA's QATS contractor (CB&I 2013b). QC results for analyses performed in 2013 are in the process of being reviewed by CB&I and will be included in the next revision of this report. The following sub-sections summarize the overall conclusions from the 2007-2012 QC summary report.

12.4.1 Field Quality Control Samples

A variety of different types of field-based QC samples have been collected as part of investigations conducted at OU3. The investigation-specific SAP/QAPPs specify the types and frequency of field QC samples that were to be collected as part of each investigation. The types of field QC samples collected differ by media type, as follows:

- Lot blanks – air
- Field blanks – air, water
- Field duplicates/splits – air, water, soil, duff, tree bark
- Equipment rinsates - groundwater

A detailed review of the field QC sample results is provided in CB&I (2013b) and summarized briefly below.

Lot Blanks

A total of 14 air cassette lot blanks were analyzed by TEM. No asbestos structures were observed in any of the lot blanks analyzed. On this basis, the cassette lots were utilized for the ambient air and ABS programs. However, it was noted that no lot blanks have been analyzed since 2010 (CB&I 2013b); it is presumed that the four air samples collected during the Kootenai River recreational visitor ABS study in 2012 utilized filters from lots utilized in earlier studies (e.g., the 2010 studies), but this is not known for certain.

Field Blanks

A total of 33 air field blanks and 53 water field blanks were collected from 2007 to 2012 and analyzed by TEM. LA was detected in three water field blanks[21] (P1-00257, P5-10028, and P5-20103) suggesting that there may have been potential contamination introduced during sample collection and/or analysis. Field blank P1-00257 was collected on 10/18/2007; however, there were no field samples associated with this field blank. Based on these results, it is concluded that contamination of air samples and water samples as a consequence of field collection and analysis methods is not of concern.

Field Duplicates/Splits

A total of 84 field duplicates and 4 field splits were collected from 2007 to 2012 and analyzed by TEM. The TEM results for the original and field duplicate/split samples are compared using the method for comparison of two Poisson rates described by Nelson (1982), based on a 90% confidence interval. Because field duplicate/split samples are expected to have inherent variability that is random and may be either small or large, there is no quantitative requirement for the agreement of field duplicates/splits. Results provide information on the magnitude of this variability and its effect on data interpretation.

The evaluation of field duplicates/splits suggests that the reproducibility of TEM results for air samples is good, but the reproducibility of water, tree bark, and duff TEM results (even within a small sampling scale) is difficult due to the inherent heterogeneity within the medium. In general, when field duplicate/split samples were statistically different from the original sample, concentrations were usually within a factor of about 3 for water samples and within a factor of about 10 for tree bark and duff samples.

A total of 31 field duplicates for soil-like media were collected from 2007 to 2012 and analyzed by PLM-VE. Field duplicate results are ranked as concordant (in agreement) if both the original sample result and the field duplicate result report the same semi-quantitative PLM-VE bin. Results are ranked as weakly discordant if the original sample result and the field duplicate result differ by one semi-quantitative bin (e.g., Bin A vs. Bin B1). Results are ranked as strongly discordant if the original sample result and the field duplicate result differ by more than one semi-quantitative bin (e.g., Bin A vs. Bin B2).

The evaluation of field duplicates for soil-like media shows that most field duplicates (~80%) were concordant with the original sample results. When results were discordant, they were only weakly discordant (i.e., within one bin). These differences may be due to analytical variability, but might also arise from authentic heterogeneity between the samples.

[21] The CB&I (2013b) report identifies four field blanks with structures, but the originally reported result for sample P5-10014 was found to be in error (see Section 12.6) It is possible that the results for sample P5-20028 are also in error, but this was not able to be confirmed.

Equipment Rinsates

A total of 5 equipment rinsates were collected in Phase II Part B as part of groundwater collection efforts and analyzed by TEM. LA was detected in one equipment rinsate (concentration of 0.35 MFL based on total LA). This indicates that the decontamination procedures applied were not effective and that LA may have been introduced into the groundwater samples due to cross-contamination. Two groundwater field samples (P2-00780 and P2-00781) were collected on the same day with this equipment rinsate; total LA concentrations in these two field samples ranged from non-detect to 0.1 MFL based on total LA. Due to the contamination in the equipment rinsate, these two samples were FB-qualified.

12.4.2 Preparation Laboratory Quality Control Samples

The preparation laboratory QC samples are used to ensure that the preparation techniques utilized to process soil-like samples did not introduce potential contamination and to evaluate variability associated with preparation techniques.

There are two types of preparation laboratory QC samples that were evaluated at the Libby site: preparation blanks (including both grinding blanks and drying blanks) and preparation duplicates. A detailed review of the preparation laboratory QC sample results is provided in CB&I (2013b) and summarized briefly below.

Preparation Blanks

All 48 preparation blanks that were inserted along with OU3 soil-like samples were ranked as non-detect (Bin A) by PLM-VE. These results show that the drying and grinding preparation procedures utilized within the CSF and Troy SPF did not introduce LA contamination.

Preparation Duplicates

From 2007 to 2012, a total of 34 preparation duplicates were prepared by the CSF or Troy SPF and analyzed by PLM-VE. Comparison of the preparation duplicate results with the paired original field sample results helps to evaluate the variability that may occur during sample preparation and analysis. Similar to field duplicates, preparation duplicates are ranked as concordant if both the original sample results and the preparation duplicate results display the same semi-quantitative classification. Most (~75%) of the preparation duplicates were ranked as concordant. When results were discordant, they were only weakly discordant. These results suggest that the PLM-VE results are generally reproducible and reliable and are not greatly influenced by differences in laboratory preparation and analysis techniques.

12.4.3 Analytical Laboratory Quality Control Samples

TEM

The laboratory QC requirements for TEM analyses at the Libby site are patterned after the requirements set forth by NVLAP, and include:

- Laboratory blanks
- Re-preparations
- Recounts (i.e., recount same, recount different, and verified analyses)
- Inter-laboratory analyses

A detailed review of the laboratory QC analysis results is provided in CB&I (2013b) and summarized briefly below.

Laboratory blanks. No asbestos structures were observed in any laboratory blank samples analyzed by TEM. These results indicate that the filter preparation and analysis procedures utilized within the analytical laboratories did not introduce asbestos contamination.

Repreparations. A total of 35 repreparation TEM analyses have been performed for OU3 samples analyzed from 2007 to 2012. Repreparation analyses are compared to the original analysis using the ratio method for statistical comparison of two Poisson rates recommended by Nelson (1982), based on a 90% Poisson confidence interval (CI). With the exception of five samples (14%), repreparation results were not statistically different from the original results. Repreparation analyses were statistically different for two surface water repreparation analyses (collected as part of the Phase II-A investigation), two pore water samples (collected as part of the Phase V-B investigation), and one tree bark sample (collected as part of the Phase IV-A investigation). These results show that LA concentrations in air and duff reported in the OU3 investigations have acceptable reproducibility and that TEM analytical precision is not likely to be impacted by filter preparation methods. But, the reproducibility of water samples is ranked as poor, which highlights the inherent difficulties in sampling this medium.

Recounts. More than 250 GOs and 800 structure pairs were re-examined as part of recount analyses for OU3 from 2007 to 2012. Recount analyses were compared with the original analysis on a GO-by-GO and structure-by-structure basis. GO concordance is evaluated based on a comparison of total structure count. Structure concordance is evaluated based on a comparison of the assigned mineral classification and recorded structure dimensions. The total structure counts matched for about 90% of all GOs, which ranks as acceptable concordance (per Libby laboratory modification LB-000029). When the same structure was observed and recorded, there was 100% agreement on the assigned mineral class and good agreement (91% for length; 98% for width) on the recorded structure dimensions. These results indicate that there is good result reproducibility between TEM analysts within the same laboratory.

Inter-laboratory Analyses. More than 90 GOs and 400 structure pairs were re-examined as part of inter-laboratory analyses for OU3 from 2007 to 2012. Inter-laboratory analyses are special type of recount analysis, in which GOs are re-examined by a different laboratory than who performed the original analysis. Inter-laboratory analyses are compared in the same way as recount samples (described above). The total structure counts matched for only about 55% of all GOs, which ranks as poor concordance (per Libby laboratory modification LB-000029). When the same structure was observed and recorded, there was 98% agreement on the assigned mineral class for paired structures, which is ranked as acceptable (per Libby laboratory modification LB-000029). When mineral class differences were noted, it was usually related to differences in classification of "close call" non-asbestos material [NAM] (e.g., pyroxene). Although there was good agreement (94%) between laboratories on the recorded structure width, several discrepancies in recorded structure length were noted, and overall concordance was poor (71%). The TEM inter-laboratory analyses indicate there are differences structure identification and recording procedures between the TEM laboratories corrective action would be useful in achieving better agreement and reducing uncertainties due to between-laboratory differences.

PLM

Three types of laboratory-based QC analyses are performed for PLM-VE, including laboratory Duplicates (both self-checks and cross-checks), inter-laboratory analyses, and the performance evaluation (PE) standard analyses.

Laboratory Duplicates. A total of 52 PLM-VE laboratory duplicate analyses have been performed for OU3 samples analyzed from 2007 to 2012. Comparison of the laboratory duplicate results with the paired original field sample results helps to evaluate the variability that may arise during the PLM analysis. Similar to preparation duplicates, laboratory duplicates are ranked as concordant if both the original sample results and the laboratory duplicate results display the same semi-quantitative classification. Nearly all of the laboratory duplicates were ranked as concordant (only one analysis ranked as weakly discordant). These results indicate that the PLM-VE results are generally reproducible and reliable and are not greatly influenced by differences in analysis techniques within a PLM laboratory.

Inter-laboratory Analyses. A total of 19 PLM-VE inter-laboratory analyses have been performed for OU3 samples analyzed from 2007 to 2012. In general, the reproducibility of results between PLM-VE laboratories was poor for OU3 samples, with only about half of all inter-laboratory analyses ranked as concordant and many samples ranked as weakly discordant. The PLM-VE inter-laboratory analyses suggest that there are differences in methods and procedures between the PLM laboratories and corrective action is needed to achieve better agreement and reduce analytical uncertainties.

PE Standard Analyses. Libby-specific PE standards for soil have been created for use at the Libby site. These PE standards were created by spiking soil with known quantities of LA

obtained from the mine. A total of 40 PE standard analyses have been performed by the PLM laboratories that support OU3. About 80% of all PE standard analyses were concordant with the expected bin classification (as determined from the nominal LA level in the PE standard). When results were discordant, they were usually weakly discordant; however, there were two strong discordances noted for the highest PE standard, with reported results being biased low. These results demonstrate that PLM-VE results are generally accurate but there are inherent uncertainties associated with reported binned results.

12.5 Data Management Quality Assurance Activities

12.5.1 Database

Application

The master OU3 project database is a Microsoft Access® relational database that has been developed specifically for OU3. Due to the nature of asbestos analysis and other data reporting requirements, the database has been developed iteratively, expanding in its capabilities (and complexity) as project-specific needs have evolved. In addition to providing new functionality, as needed, enhancements have been made to accommodate data user needs and to incorporate various automated QA/QC procedures to improve data integrity.

Because data are continually being generated as a result of ongoing sampling and analysis at OU3, the project database is dynamic. Each day, new sample, analysis, and results records are added and records are corrected, as appropriate. As a result, any database-generated queries, tables, figures, maps, and reports provide only a "snapshot" of the database on the day the output was created. **Appendix A** provides a snapshot of the OU3 project database as of January 20, 2015. This snapshot was used to prepare all data summaries included in this report.

Administration and Security

Day-to-day operational control of the OU3 project database is under the control of EPA's contractor, CDM Smith, including physical and network security, access rights, and data backup. The OU3 project database is kept on the CDM Smith server in Denver, Colorado. Incremental backups of the CDM Smith server are performed daily Monday through Friday, and a full backup is performed each Saturday. Access to the server is restricted to approved CDM Smith personnel only.

Data Entry Processes

The OU3 project database has a variety of built-in QC functions that improve accuracy of data entry and help maintain data integrity. For example, field data entry forms utilize drop-down menus whenever possible. Drop-down menus allow the data entry personnel to select from a set of standard inputs. The use of drop-down menus prevents duplication and transcription

errors and limits the number of available selections (e.g., valid media types). In addition, the project data allows a unique sample ID to only be entered once, thus ensuring that duplicate records cannot be created.

As noted above, the analytical laboratories are required to transmit results using Libby-specific electronic data deliverable (EDD) spreadsheets. Each EDD contains a variety of built-in QC functions that improve the accuracy of data entry and help maintain data integrity. For example, data entry forms utilize drop-down menus whenever possible to standardize data inputs and prevent transcription errors. In addition, many data input cells are coded to highlight omissions, apparent inconsistencies, or unexpected values so that data entry personnel can check and correct any errors before submittal of the EDD. These spreadsheets also perform automatic computations of analytical sensitivity, dilution factors, and concentration, thus reducing the likelihood of analyst calculation errors.

The transmitted EDDs are uploaded directly into the OU3 project database using upload queries in Microsoft Access® designed specifically for each type of EDD, which avoids potential errors related to manual entry of the results. Each upload query performs several integrity checks to ensure that records are consistent and complete prior to uploading the analytical data. If issues are identified, the analytical EDD will not be uploaded until they are rectified.

12.5.2 Non-Asbestos Data Validation

All data on the concentration of non-asbestos chemicals in surface water, sediment, soil, mine waste materials, and ground water were validated in accordance with EPA's Contract Laboratory Program (CLP) *National Functional Guidelines (NFGs) for Evaluating Organic Analyses* and *NFGs for Evaluating Inorganic Analyses*, modified for the methods used at OU3.
In brief, all non-asbestos data were evaluated based on the following parameters:

- Data Completeness
- Holding Times
- Gas Chromatography/Mass Spectroscopy Instrument Tune
- Calibrations
- Blanks
- Surrogate Recovery
- Matrix Spike/Matrix Spike Duplicates
- Laboratory Control Samples
- Internal Standards (if applicable)
- Field Duplicates (if applicable)
- Compound Identification
- Compound Quantitation and Reporting Limits
- System Performance
- Other Laboratory QC Specified by the Method
- Overall Assessment of Data

If QC criteria were not met, samples were qualified as follows:

R: Reported value is "rejected." Resampling or reanalysis may be necessary to verify the presence or absence of the compound.

J: The associated numerical value is an estimated quantity because the QC criteria were not met.

UJ: The reported quantitation limit is estimated because QC criteria were not met. Element or compound was not detected.

NJ: Estimated value of a tentatively identified compound. (Identified with a CAS number.) *Organics analysis only.*

U: The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

NR: Result was not used from a particular sample analysis. This typically occurs when more than one result for a compound is reported due to dilutions and reanalyses.

The non-asbestos data validation was performed by EPA subcontractor, TLI Solutions, Inc. (TLI). A summary of the non-asbestos data validation is provided in CB&I (2013c). In brief, inorganic, organic, and/or radiochemistry data for a total of 651 water and soil-like media samples in 29 sample delivery groups were reviewed by TLI. The OU3 project database (provided in **Appendix A**) includes all assigned data validation qualifiers. Any samples that were R-qualified (rejected) by the data validator should be excluded from use as the results are not reliable.

12.5.3 Asbestos Data Verification

Prior to the preparation of any data summary reports, a cursory data review is performed on any applicable data in the OU3 project database to identify data omissions, unexpected values, or apparent inconsistencies. Because analytical laboratories that utilize Libby-specific EDD spreadsheets, data checking of reported analytical results begins with automatic QC checks that have been built into these spreadsheets. In addition to these automated checks, as dictated by the governing investigation-specific SAP/QAPP, a more thorough data verification evaluation is also performed to ensure the consistency and quality of reported data.

Asbestos data verification includes checking that results have been transferred correctly from the original hand-written, hard copy field and analytical laboratory documentation to the OU3 project database. This data verification process utilizes Libby-specific SOPs developed to ensure

TEM and PLM results and field sample information in the OU3 database are accurate and reliable:

- EPA-LIBBY-09 – *SOP for TEM Data Review and Data Entry Verification* – This SOP describes the steps for the verification of TEM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.

- EPA-LIBBY-10 – *SOP for PLM Data Review and Data Entry Verification* – This SOP describes the steps for the verification of PLM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.

- EPA-LIBBY-11– *SOP for Field Summary Data Sheet (FSDS) Data Review and Data Entry Verification* – This SOP describes the steps for the verification of field sample information, based on a review of the FSDS form, and verification of the transfer of results from the FSDS forms into the project database. An FSDS review is performed on all samples selected for TEM or PLM data verification.

The goal of data verification is to identify and correct data reporting errors. The frequency of data verification is specified in each investigation-specific SAP/QAPP; typically, a minimum of 10% of sample and analysis results are verified.

There have been several data verification efforts performed in association with each OU3 investigation. Detailed results of data verification efforts and data quality conclusions are provided in the OU3 data verification summary report (see **Appendix E**). In brief, most of the issues identified during these data verification efforts were non-critical in nature, meaning that they were typographical errors and inconsistencies that were not expected to influence LA results and data interpretation. The frequency of critical errors (i.e., those that could influence LA results and data interpretation) was generally low. Error frequencies tended to be higher following particular programmatic changes in laboratory methods and data reporting requirements and at the beginning of sampling investigations.

All issues identified during the various OU3 data verification efforts were submitted to the field teams and/or analytical laboratories for resolution and rectification. All tables, figures, and appendices (including the OU3 project database provided in **Appendix A**) generated for this report reflect corrected data.

12.5.4 Asbestos Data Validation

Unlike asbestos data verification, where the goal is to identify and correct data reporting errors, the goal of asbestos data validation is to evaluate overall data quality and to assign data qualifiers, as appropriate, to alert data users to any potential data quality issues.

Until recently, there have been no formal data validation guidelines for asbestos. Thus, data validation efforts were performed by EPA technical contractors following the completion of each investigation and consisted primarily of a review and assessment of field and laboratory ROM forms, field QC data (e.g., field duplicates, field blanks), and laboratory QC data (e.g., recounts, repreparations) to evaluate potential data quality issues with respect to result precision, accuracy, representativeness, completeness, and comparability. No review of instrument calibration or control standard data was performed, as this type of information was included in the regular NVLAP certification process.

In late 2011, EPA released a draft of the *NFG for Asbestos Data Review* (EPA 2011b). These guidelines include review criteria and specific data qualifiers for validation of TEM, PLM, and phase contrast microscopy (PCM) data. CB&I developed Libby-specific SOPs for data validation of asbestos datasets based on the draft asbestos NFGs. In 2013, CB&I performed a formal data validation of asbestos results for OU3 investigations conducted from 2007 to 2012; a detailed summary of this data validation effort is summarized in CB&I (2013b). In 2014, CB&I performed a formal data validation of asbestos results for OU3 investigations conducted in 2013; a detailed summary of this validation effort is summarized in CB&I (2014). The conclusions of these reviews are summarized below.

A total of 360 field samples (5%) from 30 different laboratory jobs analyzed by five different laboratories between 2007 and 2012 were selected for validation. For 2013, data validation was performed for 51 field samples (5%). Samples for validation were selected randomly, choosing samples that were representative across laboratory, analysis method, and media.

Very few OU3 asbestos data were qualified. For the 2007-2012 validation effort, only one laboratory QC analysis (recount different) was assigned a J-qualifier; no other OU3 analyses required qualification. Data for these this analysis was qualified due to the failure of the laboratory to perform and/or document daily calibration activities. Although several samples were affected by the lack of a daily calibration, they were not qualified due to the submission and review of other supporting laboratory documentation. For the 2013 validation effort, four tree bark samples were J-qualified due to the failure to meet the required quarterly frequency for the k-factor calculation. The OU3 project database (provided in **Appendix A**) includes all assigned data validation qualifiers.

12.6 2012 Water Result Discrepancy Evaluation

A review of unexpected results for water samples collected from OU3 during the Phase V, Part A and Part B investigations indicated that samples were likely misidentified in the laboratory while being processed. All of the samples in question were prepared and analyzed by EMSL-Libby in the spring of 2012. A detailed summary of the discrepancies identified, steps taken to resolve these discrepancies, and resolutions to ensure that reported results in the project

database are accurate is provided in a technical memorandum prepared by the QATS contractor (CB&I 2013a).

This discrepancy evaluation concluded that filter mix-ups occurred at EMSL-Libby (likely during ozonation/UV treatment). The largest mix-up appears to be associated with the set of filters that were prepared during Round 3 of the Phase V, Part A (Kootenai) sampling effort. However, other filter mix-ups outside of this timeframe were also noted. This discrepancy evaluation also showed that there were differences between the different EMSL laboratories in the identification and recording of LA structures in water samples from OU3; the magnitude of the differences in the reported water concentrations are usually not large (within a factor of 2-3).

As presented in this memorandum, the following recommendations were made:

- Because the analyst erroneously utilized PCM (not TEM) counting rules when performing the rapid TAT analyses for Kootenai River surface water samples, all rapid TAT results reported as part of the Phase V, Part A study should be disregarded. Only standard TEM results should be retained.

- For samples where the re-analysis (performed by EMSL-Cinnaminson) confirmed that a filter mix-up occurred (i.e., P5-10014, P5-10015, P5-10017, P5-10018, P5-20325, and P5-20326), the original EMSL-Libby results will be rejected and replaced by the EMSL-Cinnaminson results.

- For all other 2012 water samples (Phase V, Part A and Part B), the EMSL-Libby result will be retained. However, data users should be aware of the added uncertainty in these results due to between-laboratory differences in TEM counting and recording procedures.

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